



## **COCOA AND CHOCOLATE**









# COCOA AND CHOCOLATE

## THEIR CHEMISTRY

AND

## MANUFACTURE

*REVISED AND ENLARGED*

BY

R. WHYMPER

SECOND EDITION

*WITH 16 PLATES AND 88 TEXT-FIGURES*



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## PREFACE

### TO THE FIRST EDITION

It has been the object of the author, in producing this work, to provide a standard book of reference dealing with cacao from its growth till manufactured into cocoa or chocolate.

For the first time, the chemical composition of cacao has been traced from the fresh beans through the various stages of manufacture, so that the changes which occur during fermentation, drying, roasting, etc., may be followed.

The most recent methods of analysis have been discussed, and the author has only recommended those which he himself has found to give accurate and consistent results.

Throughout the book, the word *cacao* has been used for the raw material, that is to say, for the beans or their parts from which nothing has been taken or which have had nothing added to them; for example, after roasting and husking the *cacao* beans, the *cacao* nibs are expressed, and the fat is removed with the formation of *cocoa* powder. The word *cacao* is also used before "butter," signifying the fat extracted from the *cacao* bean, owing to the possibility of confusion with coconut fat (obtained from *Cocos nucifera*).

I am glad of this opportunity to thank those who have assisted me in this work, and especially Mr. G. H. Davis and Mr. W. P. Paddison. My thanks are also due to Messrs. Baker & Sons, Ltd., of Willesden, and Messrs. Bramigk & Co., for their permission to reproduce photographs of their most up-to-date machinery.

R. W.

DETTON HILL,  
SURREY.

1912

## PREFACE

### TO THE SECOND EDITION

SINCE the date of the first publication of this work, much water has flowed under London Bridge. It is difficult to think back to the good old days when necessities and commodities were cheap, and the public appetite was normal. It was then perfectly possible to say what a machine would cost, and to predict the day of delivery. It was possible to foresee, to some extent, the trend of public fancy, and to guide its taste in certain directions.

To-day, none of these things is possible. The chocolate industry is immense and growing very rapidly. There are also, on the one hand, high prices ruling labour and the cost of materials, and a general shortage of necessary ingredients whilst, on the other hand, the increasing demand for sweet things does not ease the situation. Even now, it is not possible to say how long the public will continue to pay the very high prices for cocoa and chocolate, nor is it possible to predict whether, in the immediate future, the market value of sugar and cacao will go up or down. The even distribution of these ingredients over the world, according to the demand, has not yet taken place, and the consumption of sweet things, though still very great, must fall sooner or later, if prices continue to rise.

Huge amalgamations have become the order of the day and science has been called upon by the chocolate industry to assist in the manufacture of a product that was once a luxury, yet cheap, but which is now an expensive necessity.

I have every reason to believe that this work, originally published at a critical time in the history of the industry, has served a useful purpose in putting before the manufacturer the ways and means of securing a desired product, and in providing scientific reasons for the development of flavour and consistency. At any rate, its publication was accorded a very cordial reception, and I have the satisfaction of knowing that a new edition has been demanded, and is awaited with some impatience.

An industry that has grown with such rapidity as that producing cocoa and chocolate must, necessarily, develop and change. New ideas for manufacture have arisen from the necessity for both increased output and labour-saving devices. Generally speaking, the quality of cocoa and chocolate was very good in Europe before 1914, and the manufacturer devoted much time, money and thought to his product. Then came the war and a greatly increased demand for cocoa confections, a shortage of ingredients, and labour difficulties. It was inevitable that radical changes should be introduced into the factory. But what then of the quality of the product?

Speaking as one who consumed in those dark days more chocolate than ever previously in his lifetime, I am aware that, at first, I was severely critical, but later, with palate possibly spoiled by too much tobacco, whiskey, bully-beef and biscuits, almost any chocolate was chocolate, sweet and palatable. This I believe to have been the experience of most people, though, often enough (and I would not attribute the depreciation of the palates of others always to those reasons given in my own case), it was mere shortage of chocolate on the market that dulled the powers of discrimination. In any case, it is certain that any chocolate on the market was saleable in those sugarless days and was accounted a luxury.

In returning to more normal times, the manufacturer

cannot afford to ignore quality as undoubtedly he was compelled to do after the dog days of 1914. Competition and a more fastidious public have again to be faced. Some changes in the factory have come to stay, and they are discussed in later pages, whilst the influence of these changes on quality is also considered as far as possible.

I have had exceptional opportunities of studying the new side of the chocolate industry, as I have returned to face the problems with a fresh outlook and have also had the advantage of visiting the United States of America in the latter part of 1919 and in early 1920.

The American is not hampered by precedent and is ever prepared to try new ideas. Moreover, he is a "quantity" specialist and, very naturally, wide-awake to any measures that will increase output. Further than this, at the time of writing, his output of chocolate is much below that demanded by the public. America, therefore, may be said to be in much the same condition now as this country was during the war, with the exception that the American public has never been educated to the standard of chocolate demanded by the European. The American manufacturer can, then, put upon the market and readily sell products that would not be considered "best quality" in Europe, and it is my opinion that his reputation, on this side, has suffered adversely from the chocolates he has thought fit to send us during our famine.

This condition, I feel certain, will change in the very near future. Chocolate factories are springing up like mushrooms all over America, whose soil, moistened by the beer and wine that has been spilt, and warmed by the enthusiasm of "Pussyfoot," has become particularly suitable for upstart industries of this kind. Doubtless, posterity will bless the accomplishment of "prohibition" and, incidentally, will reap the advantage of better chocolate, for competition will be very severe.

Chocolates made in America and Europe to-day are, respectively, as different as chalk from cheese. Perhaps the European taste is that of a *blasé* epicure; perhaps the American palate is uncultured. I will not take it upon myself to pass judgment. But, and this I can say with some assertion, chocolate made in the good old-fashioned way, with careful consideration paid to every detail, to ingredients and to processes of manufacture, is still unequalled for delicacy of flavour and aroma by any chocolate manufactured by short-cut methods.

I am fully aware of the errors and shortcomings of the first edition of this work. They were inevitable, seeing that I worked almost single-handed, but they have been corrected and improved, respectively, in the present edition, so far as in me lies. Indeed, most of the work has been re-written, and much, too, has been added, a labour which, at this time, I could not have accomplished alone.

On the practical side, I have received the most able assistance from Mr. A. B. Bradley and Mr. W. P. Paddison, the former in connection with the chemistry of cocoa and chocolate, the latter in providing photomicrographs. Mr. Bradley, by his numerous experiments and analyses, has acquired more reliable information than I could possibly have hoped to secure by myself. His work on sugar is, in my opinion, particularly valuable and points out the correct direction in which to turn to cheapen chocolate, without lowering its quality. In this direction, we have worked together for many years, with the result that, with the kind and most extensive co-operation of Mr. John Carr—without whom our work would have been abortive,—a new and valuable asset has been added to the chocolate industry. I am not a little indebted to Mr. W. J. Magenis in the compilation of this edition. In the most indefatigable manner, he has hunted down published information on the subjects in hand, often finding it in most obscure



## PREFACE TO THE SECOND EDITION

journals, which, otherwise, would have remained unknown to me.

Mr. W. L. Dubois, formerly of the United States Bureau of Chemistry, who has sent me private communications concerning, and the analyses of, many American chocolates, has also rendered me great service, at the same time adding much to the statistical value of this book. To him I am most grateful, and I wish him luck in his new undertaking where he will be able to continue his researches in the chocolate industry.

From Messrs. Joseph Baker, Sons and Perkins, Ltd., both in London and New York, I have received very great assistance, and it is gratifying to me to be able to record my indebtedness to this firm, which, though absorbed from 1915 until the armistice in making shells and other munitions to assist in bringing the war to a successful conclusion, can now boast of providing machinery, equalled by few and surpassed by none, for use by manufacturers of cocoa, chocolate and confectionery.

Though the superiority of some German machinery for the industry, especially of mills and granite rolls, before the war, cannot be denied, it is certain that the British manufacturer can hold his own to-day. His business is difficult and specialised, and it is particularly pleasant to observe that the engineering firms in this country, making this class of machinery, have taken advantage of their opportunities to pass the foreign competitor.

Prior to 1914, Messrs. Lehmann, of Dresden, produced some of the finest chocolate and cocoa machinery in the world, and many examples of their excellent work stand to their credit in the best factories in Europe and America. It is rather early to pass judgment on the machinery that Germany is turning out to-day, but, from the orders in the houses of Mr. Weygandt and Messrs. Bramigk & Co., Messrs. Lehmann's agents in New York and London, respec-

tively, it is clear that the same thoroughness and good workmanship, that characterised that firm's machines of old, are expected by their former clients and adherents. I am indebted both to Mr. Weygandt, and to Mr. Hillary of Messrs. Bramigk & Co., for many kindnesses and, especially, for permission to show illustrations of machines for the excellence of which I can vouch.

Messrs. Savy Jeanjean et Cie., Courbevoie, near Paris, also readily responded to my request for illustration blocks of their "Enrober" and "Caramelising" machines, the former of which is, unquestionably, the best automatic covering machine on the market. I welcome this opportunity of expressing my appreciation of their ready assistance.

To many others, too numerous to mention by name, to the critics of the first edition of this work, who have been many, and to those engaged in the manufacture of cocoa and chocolate, I tender my warmest thanks for the assistance, criticisms and courtesies which have so greatly helped me to bring this work up-to-date.

R. W.

SURBITON,  
SURREY.

# CONTENTS

	PAGES
PREFACES . . . . .	v—xi
INTRODUCTION . . . . .	xvii—xxi

## PART I

### HISTORY, BOTANY AND AGRICULTURE OF CACAO

CHAPTER		
I.	History and Growth of the Cacao Industry . . . . .	3—17
II.	Statistics of the Cacao Industry . . . . .	18—30
III.	Botany and Nomenclature . . . . .	31—37
IV.	The Agriculture of Cacao—A. Selection of Site, Soil, Manure and Seed . . . . .	38—47
V.	The Agriculture of Cacao—B. Planting, Diseases, Pruning and Picking . . . . .	48—57
VI.	Fermentation of the Beans . . . . .	58—76
VII.	Preparation of the Beans for Market—Washing, Drying, Finishing or Polishing; <i>Résumé</i> of Chapters IV. to VII. inclusive—Importance of Natural Conditions, and of Processes of Fer- mentation and Curing in Determining Quantity and Quality of the Finished Cacao . . . . .	77—89
VIII.	Characteristics of the Principal Kinds of Commer- cial Cacao Beans . . . . .	90—118
IX.	Composition of Cacao Beans . . . . .	119—125

## PART II

### MANUFACTURE OF CHOCOLATES AND COCOA POWDERS

X.	Cleaning, Sorting and Grading Cacao, prior to Roasting . . . . .	129—134
XI.	Roasting of Cacao—Analyses of Beans, Nibs and Shells, before and after Roasting . . . . .	135—145
XII.	Nibbing, Husking and Winnowing the Roasted Cacao —Analyses of Nibs and Shells, and their Pro- portions in the Roasted Bean . . . . .	146—161
XIII.	Milling—Preparation of Cocoa Powder—Expression of Cacao Butter—"Soluble" Cocoa Powders . . . . .	162—183

# CONTENTS

xiii

CHAPTER	PAGES
XIV. Analyses of Cocoa Powders, "Soluble" Dietetic, Proprietary, etc.	184—200
XV. Preparation of Chocolate—Mixing—Refining .	201—216
XVI. Short-Cuts to the Manufacture of Chocolate—Influence of Method on Quality .	217—236
XVII. Moulding Chocolate Tablets, Croquettes, Neapolitans and Fancy Shapes: Tapping, Cooling, Wrapping .	237—260
XVIII. Analyses of Plain Eating-Chocolates and Chocolate Powders .	261—275
XIX. Recipes for Making Chocolate—Plain and "Fondant" Chocolates, and Chocolate Powders .	276—289
XX. Milk Chocolates and Milk Chocolate Powders—Analyses and Recipes—Nut Chocolates, etc. .	290—306
XXI. Milk Chocolate and "Chocolat Fondant"—Methods of Manufacture .	307—323
XXII. Covering-Chocolate—Manufacture and Analyses—Use of "Enrober," etc. .	324—340
XXIII. Aroma and Flavour due to Variety of Bean, Process of Manufacture, and Flavouring Materials, such as Vanilla, Spices, Essences, etc. .	341—353
XXIV. Nutritive Value and Digestibility of Cocoa Preparations .	354—367

## PART III

### CHEMISTRY OF CACAO.

#### A SURVEY OF THE COMPONENTS OF CACAO, COCOA AND CHOCOLATE.

#### B. METHODS OF ANALYSIS

##### A.

XXV. Survey of the Component Parts of Cacao Beans—Husk .	371—399
XXVI. Survey of the Component Parts of Cacao Beans—Nibs .	400—416
XXVII. Cocoa Powders and Chocolates, and their Component Parts .	417—433
XXVIII. Microscopical Examination of Cacao, Cocoa, Chocolates, etc. .	434—447

CHAPTER	PAGES
XXIX. Methods of Analysis: Fat—Determination of Physical and Chemical Constants of Cacao Butter and its Substitutes . . . . .	448—478
XXX. Methods of Analysis: Moisture—Mineral Matter (Soluble, Insoluble, Alkalinity of)—Cold-Water Extract—Fibre (Crude Fibre, Cellulose, Pectosans)—Cacao-Red—Xanthine Bases (Theobromine, Caffeine)—Organic Acids (Tartaric, Acetic, Oxalic) . . . . .	479—505
XXXI. Methods of Analysis: Reducing Matters—Total Nitrogen and Albuminoids—Starch, Cane Sugar and Milk Sugar—Cacao Matter . . . . .	506—532
XXXII. Added Husk—its Detection in Cocoa Preparations . . . . .	533—548
APPENDIX . . . . .	549—552
BIBLIOGRAPHY . . . . .	553—555
INDEX . . . . .	557—568

## ILLUSTRATIONS

PLATE		TO PAGE PAGE
	Cacao Pods . . . . . <i>Frontispiece</i>	
I.	Theobroma Cacao . . . . .	31
II.	A well-watered Cacao Plantation, Trinidad . . . . .	30
III.	Cacao Trees shaded by "Madre de Cacao," Trinidad . . . . .	49
IV.	Picking the Cacao Pods, Trinidad . . . . .	53
V.	Carrying the Cacao Pods to the Slicing Heaps, Trinidad . . . . .	57
VI.	A Heap of Cacao Pods ready for Slicing, Trinidad . . . . .	61
VII.	Opening the Cacao Pods, Trinidad . . . . .	65
VIII.	Sweating Box, Trinidad . . . . .	69
IX.	Artificially Drying Cacao, Trinidad . . . . .	75
X.	Cacao Beans Drying in the Sun, Trinidad . . . . .	79
XI.	"Claying" Cacao, Trinidad . . . . .	81
XII.	"Dancing" the Cacao, Trinidad . . . . .	85
XIII.	Sorting Cacao Beans on a Drying Floor, Trinidad . . . . .	91
XIV.	Bagging Cacao for the Local Market, Trinidad . . . . .	99
XV.	Bagging Cacao Beans for Shipment, Trinidad . . . . .	107
<b>FIGURE</b>		<b>PAGE</b>
1.	Cacao Bean Sorting and Cleaning Machine, with Sorting Band . . . . .	133
2.	Cacao Bean Roaster, for Open Fire . . . . .	143
3.	Improved Gas Cacao Roasting Machine. Externally heated, either by town or "producer" gas . . . . .	144
4.	New and improved Nibbing, Husking and Winnowing Machine. Front view . . . . .	152
5.	New and Improved Nibbing, Husking and Winnowing Machine. Back view . . . . .	153
6.	Cacao Germ-separating Machine . . . . .	154
7.	Double Cacao Grinding Mill, with 33-inch and 36-inch Top Runner Stones . . . . .	164
8.	Triple Cacao Grinding Mill . . . . .	165
9.	Triple Cacao Grinding Mill, with 33-inch and 36-inch Top Runner Stones . . . . .	166
10.	Ten-Pot Hydraulic Cacao Butter Press, with Pump, etc. . . . .	171
11.	No. 7 Large, Patented "Melangeur," with Automatic Discharger, coupled with Three-Roll Upright Refiner . . . . .	205

FIGURE	PAGE
12. Battery of two Chambers of Porcelain Three-roll Refiners . . .	207
13. Patent Five-roll Upright Refiner, with Chilled Rolls and Spring Bearings . . . . .	208
14. Five-roll Steel Refiner, with Chilled Metal Rollers, all Water-cooled . . . . .	209
15. Combined Refiner and Water-jacketed "Melangeur" . . . . .	213
16. "Bausman Disc" Reducer and Refiner . . . . .	229
17. Cross-section and Second and Fourth-Floor Plans of "Amorphous" Sugar Plant, with Refinery System . . . . .	234
18. Elevation and Third-Floor Plan of "Amorphous" Sugar Plant, with Refinery System . . . . .	235
19. Automatic Tempering Machine for Stiff and Liquid Chocolate, Milk Chocolate, Coverings, etc. . . . .	239
20. Noiseless Shaking Table . . . . .	245
21. Patent Mould-filling Machine, combined with Improved Continuous Tapping Table . . . . .	252
22. Chocolate Moulding, Tapping and Cooling Installation . . . . .	254
23. Weighing and Moulding Machine, for all classes of Chocolate . . . . .	256
24. Large Size, New Design "Conche" Machine . . . . .	322
25. Steam-jacketed Chocolate Mixing and Kneading Machine . . . . .	330
26. "Champion" Coating Machine . . . . .	332
27. The "Enrober" Covering Machine . . . . .	335
28. The "Enpholda" Automatic Covering Machine, with Cooling System . . . . .	338
29. "Thermos" Caramelising Machine . . . . .	345
30. "Mitscherlich" Bodies between the Lobes of the Kernel. $\times 300$ diam. . . . .	435
31. "Mitscherlich" Bodies, enlarged. $\times 1,000$ diam. . . . .	436
32. Cacao Husk, showing Bundles of Spirals. $\times 1,000$ diam. . . . .	437
33. Crushed Cacao Nibs. $\times 1,000$ diam. . . . .	438
34. Cocoa, Adulterated with Arrowroot Starch. $\times 100$ diam. . . . .	440
35. Ground Crystal Sugar. $\times 70$ diam. . . . .	444
36. Transformed Sugar $\times 70$ diam. . . . .	444
37. Chocolate Mass, made with Transformed Sugar, taken from the Mixer. $\times 70$ diam. . . . .	445
38. Chocolate Mass, as seen in Fig. 37, once Refined on Steel Refiners. $\times 70$ diam. . . . .	446

## INTRODUCTION.

"THE persons who habitually take chocolate are those who enjoy the most equable and constant health and are least liable to a multitude of illnesses which spoil the enjoyment of life." In such words does Brillat Savarin \* describe the virtues of chocolate, and, whether from the fact that cocoa preparations do actually assist in maintaining good health or for the reason that chocolate is the most pleasing confection to the palate, it is certain that the growth of the cacao industry, in the last three centuries, is little short of remarkable.

First as a beverage, then as a sweetmeat, chocolate has steadily worked its way into public favour, and it is safe to say that in no civilised country of the world is the "Prince of sweetmeats" unknown, and that, wherever civilised man has explored, travelled or sojourned, there may be found a wrapper, yellow, blue, red or white, that once contained a tablet of some well-known brand.

The popularity of chocolate is well deserved, for, besides being a sweetmeat, this confection possesses a food-value of a high order. Dr. Johnson has shown that cocoa essence contains as much flesh-forming bodies as the solids of dried milk, and Professor Forster † has compared the heat-giving power of cacao and cocoa preparations with some of our most valuable foodstuffs. Thus :

1 kilo of lean beef . . . . .	gives 1,214 calories.
1    ,,    fat beef    . . . . .	,,    3,057    ,,

\* Brillat Savarin, " *Physiologie du Goût.* "

† Professor J. Forster, " *Ueber holländische Kakao.* " Ein Beitrag zum Verständnis der Bedeutung des Kakaos als Genuss- und Nahrungs-Mittel."  
w.c.



1 litre of cow's milk	gives 672 calories.
1 kilo of hens' eggs (av. 18 eggs) •	1,678 „
1 „ husked earth-nuts	5,184 „
1 „ peas	2,710 „
1 „ white bread	2,528 „
1 „ cacao beans	5,110 „
1 „ cacao mass	5,967 „
1 „ cocoa powder	4,167 „
1 „ chocolate	4,763 „

These figures, which show the true food-value of various of our nourishing foods, explain the remarkable sustaining power of cocoa preparations, a characteristic which is recognised at the present time by our explorers, athletes, soldiers, sailors and others engaged in work or pastimes which entail great physical endurance.

The ancient Mexicans appreciated the stimulating and sustaining properties of chocolate, for Benzoni tells us that, during the time of festivities, "they used to spend all the day and half the night in dancing with only cacao for nourishment."

During the Great War, the value of cocoa preparations as food was greatly appreciated by the fighting troops. In all ranks of all the services, chocolate was used both as an emergency ration and as a luxury. It was a convenient and concentrated form of pleasant foodstuff which no expeditionary or attacking force could afford to do without, and many a man has blessed the sender of a parcel, containing chocolate, and has owed his life to that wonderful confection. Without tobacco, rum and chocolate, placed in the order of their necessity, an army in modern warfare would be defeated even with a sufficiency of guns and ammunition.

Apart from the valuable properties of chocolate as a food, there remains the all-important fact that it is pleasing to the palate. All classes, all ages, both sexes, at all times and in

all places, now eat chocolate because they like it, and not because it is a stimulating and nourishing food ; and it is this class of consumer, enabling him to produce a good, wholesome and palatable preparation within the reach of all, that appeals to the manufacturer.

“ It is fairly safe to say, however, that no single article of diet has emerged scathless from the critical survey of the dietetic reformer or his antagonist,” writes Dr. Bryce in his little handbook on “ Dietetics.” Alcohol, we know, has been the subject of much discussion, and its use is now universally recognised as the better for restriction. “ But,” continues Dr. Bryce, “ the virulent antipathy displayed by fanatical temperance reformers by reason of its use as a beverage or even as a medicine is quite misplaced, if they are themselves devotees of the ‘ cup that cheers yet not inebriates.’ For tea, coffee and cocoa all owe their undoubtedly stimulating effects to their contained active principle, viz., caffeine, or one of its allies, a drug which can be procured from any chemist’s shop and is to be found on the shelves of every dispensing physician. When used in moderation the valuable stimulating effects of these beverages is unquestionable, although it is undoubted that, just as a certain percentage of the population is poisoned by the smallest quantity of alcohol, many people display a decided idiosyncrasy for tea, coffee or cocoa. Some, indeed, exhibit a condition of chronic intoxication manifested by palpitation, breathlessness, nervousness, headache, indigestion and, in particular, neuralgia and mental and physical depression.”

Whilst this is interesting and true, and may come to many persons as “ news,” it need not cause immediate anxiety in the breasts of the consumer, or the manufacturer, of cocoa and its varieties. We are comforted to learn from the same authority that “ It is quite certain that, in the average person, the use of tea, coffee or cocoa may be continued in moderation for a lifetime without introducing any

degenerative effect on the nervous system, at any rate of a character analogous or comparable with that so well known to follow even the moderate use of alcohol."

We have occasion to refer briefly to the matter again in later pages, when the history of the growth of the cocoa and chocolate industry is under consideration, but it is of interest to observe, here, that there was some justification for the attacks made upon the beverage by Rauch, in 1624.<sup>2</sup> Further, the reason for mentioning at all, in this place, the depressing fact of the stimulating action of cacao is that the author has cause to believe that moderation is often exceeded with slight toxic effect to the consumer. The consumption of chocolate (a cocoa product with some 50 per cent. of sugar is implied) is enormous, and is increasing among the younger generation, especially among young girls. America is, perhaps, even worse than Europe at the present time in this respect—it is as well to make a virtue of necessity—and it is a matter of speculation how much of the dyspepsia and "nerves" of the youth of both continents may be attributed to the immoderate use of chocolate. In America, caffeine is already added in small quantities to some soft drinks, smaller quantities, possibly, than are present in tea, coffee or cocoa, but, nevertheless, added for the specific purpose of giving a "kick" to an otherwise sugary concoction of no individuality. It is not suggested that a drug habit would be, or is likely to be, formed by the consumption of tea, coffee, cocoa, chocolate or soft drinks containing caffeine, or its allies, naturally present or artificially added in small quantities; but it is desired to convey to the rabid food fanatics, dieteticians, alcohol-prohibitionists and others of like kidney, with which the world at present abounds, that a cocoa-drinker or chocolate-eater is not entirely guiltless of the delights of stimulation which the more sane person does not grudge him, and that the world, at large, would prefer to eat and drink what it enjoys because, or in

spite, of the fact that it is stimulated thereby, though its days shall be curtailed, rather than be legislated to consume immaculate food and drink, though its life shall be prolonged. There is also much to be said for time-honoured custom and habit.

• Did time and space allow, there are stories to be told on the romantic side of chocolate : of its divine origin ; of the bloody wars and brave exploits of the Spaniards who conquered Mexico, and who were the first to introduce cacao into Europe, tales almost too thrilling to be believed ; of the intrigues of the Court of Spain : and of celebrities who met, and sipped their chocolate in the parlours of the coffee and chocolate houses, so fashionable in the seventeenth and eighteenth centuries. But there is little room for romance in a technical work of this sort, or in these prosaic days when even the " Odour of Sanctity " has been reduced to a chemical formula. The " Food of the Gods " is now nothing but a scientific blending of certain chemical compounds, and chemists, instead of priests, are protecting the fair name of chocolate.

\* Prescott's " Conquest of Mexico," and Ogilby's " Description of America," etc.



**PART I**  
**HISTORY, BOTANY, AND AGRICULTURE**  
**OF CACAO**



## CHAPTER I

### HISTORY AND GROWTH OF THE CACAO INDUSTRY

THE commercial history of cocoa and chocolate commences with the introduction of the cacao bean into Europe in the year 1528; when Cortés, the conqueror of Mexico, returned to the court of Spain bringing with him a large collection of minerals, animals and plants to show the resources of the newly acquired country.\*

Previously to this, however, the beans of the cacao tree were much used by the Aztecs, the aborigines of Central America, who not only prepared from them the beverage known as "chocolatl," but also made use of them as a means of currency, described by Peter Martyr as "blessed money, which exempts its possessors from avarice, since it cannot be long hoarded, nor hidden underground."†

The royal revenues of Mexico were raised by imposing a tax on all the agricultural produce and manufactures of the kingdom. Among the items of the tribute furnished by different cities, Mendoza quotes: "20 chests of chocolate, 40 pieces of armour, a chest of maize, 8,000 lumps of unrefined copal, 100 copper axes, 80 loads of red chocolate, 800 'xicaras' (vessels out of which they drank chocolate), 4,000 loads of lime, 40 bags of cochineal, 20 bags of gold-dust, 200 loads of chocolate, 8,000 'handsful' of rich scarlet feathers, etc."

Torquemada has extracted the particulars of the yearly expenditure of the Mexican palace from the royal account

\* Prescott, "Conquest of Mexico."

† "De Orbe Novo," dec. v., cap. 4.



## COCOA AND CHOCOLATE

book, which included 2,744,000 fanegas of cacao, an amount approximately equal to 100,000 tons, besides 8,000 turkeys, a quantity of maize about equal to 200,000 tons, and an incredible amount of game, vegetables and condiments of every kind.

The value of the cacao beans as coins depended upon their size, and they may have been primarily looked upon as valuable from the mythical history of their origin.

The history of cacao, as given in Mexican mythology, is not without interest, for it would appear, from one of the most sacred traditions of the Indians of Mexico, that we are indebted to Divine providence for its introduction to earth. Quetzalcoatl, god of the air, was commissioned to convey to man the seeds of the "quacahault" or cacao tree which was one of the few growing in Eden for the delectation and food of the gods and the first sons of the Sun.

In the golden days of Anahuac or Mexico, when the "garden prophet" resided on earth, the land teemed with fruits and flowers without the pains of culture; a single ear of Indian corn was as much as a man could carry, and the cotton, as it grew, was dyed to the hues required by man. The air was filled with intoxicating perfumes and the sweet melody of birds, and things were always as they ought to be.

Quetzalcoatl incurred the wrath of one of the principal gods and was compelled to abandon the country, but he was so much beloved by the people of Mexico, to whom he had taught the use of metals, agriculture and the arts of government, that his return was looked for with confidence and pleasure, and a temple and altar were erected to his worship. He was said to have been tall of stature, with a white skin, long dark hair and a flowing beard; and this description, corresponding in the main with that of Cortes at the head of the Spanish invasion, prepared largely for the easy acceptance of the Spaniards into the hearts of the people. The "garden prophet" was worshipped under the name of

Votan, which signified "an adder clothed with precious or divine feathers," a highly complimentary title. Besides offering burnt incense, his worshippers were wont to lacerate their tongues, as further proof of their devotion, a proceeding which, in these days, would hardly be considered complimentary, as it might imply that they were not desirous of tasting the "Food of the Gods."

The Emperor Montezuma took no other beverage than chocolate, flavoured with vanilla or spices, and so prepared as to be reduced to "a froth of the consistency of honey."

Golden goblets, with spoons of the same metal or of tortoiseshell finely wrought, were used by Montezuma when drinking his chocolate, and the cup, after being drained, was thrown into the lake surrounding the palace. At the conquest of Mexico, a vast quantity of treasure was taken from the lake, and, judging from the number of goblets found and from the amount of chocolate prepared daily for the emperor's consumption ("fifty jars or pitchers"), it may be assumed that Montezuma was very partial to the beverage.

The beans were roasted in earthenware vessels, ground between stones and mixed with cold water, to which was sometimes added a dash of capsicum, probably as a flavouring material, though possibly as a means of increasing the devotion of the sore-tongued worshippers of Votan.

The richer people flavoured their chocolate with vanilla or spices and sweetened it with sugar or honey. The liquid was whipped into a froth, and one early historian is careful to point out the importance "of opening the mouth wide, in order to facilitate deglutition, that the foam may dissolve gradually and descend imperceptibly, as it were, into the stomach," and closes with an eulogy on the beverage so prepared.

The exact date of the introduction of cacao into Mexico from Eden is obviously rather difficult to determine, but

De Candolle,\* who has made a close study of the history of all plants of economic importance, has stated with some assertion that the tree had been in cultivation in America for three or four thousand years.

The planting of the seed seems to have called for special ceremony among the original cultivators, the shedding of the blood of man, beast or fowl being, apparently, one of the necessary operations.

Bancroft, writing of the Maya races of the Pacific, says: "Before planting the seed, they held a festival in honour of their gods, Ekchuah, Chac and Hobnil, who were their patron deities. To solemnise it they all went to the plantation of one of their number, where they sacrificed a dog having a spot on his skin the colour of cacao. They burned incense to their idols, after which they gave to each of the officials a branch of the cacao plant."

According to Ximinez, the blood of slain fowls was sprinkled over the land to be sown, a performance certainly more profitable to the soil than burnt offerings.

History does not relate of the cacao tree being of any commercial importance, however, until the conquest of Mexico, when the Spaniards found the people of the country fully appreciative of its value.

For some years the Spaniards kept the secret of chocolate preparation in their own hands, so closely indeed that we read that, during the war between Holland and Spain, the Dutch sailors, on finding cacao beans in some Spanish ships which they had captured, threw them overboard with the scornful description "*Crottes de brebis*."

The Spaniards did not long remain in undivided possession of their secret, for, in 1606, we find that one Antonio Carletti, who had himself learnt to appreciate the fine qualities of the beverage in Spain, was instrumental in bringing them to the notice of the Italians.

\* "Origin of Cultivated Plants."

## HISTORY AND GROWTH OF THE CACAO INDUSTRY 7

From this time onwards, the popularity of cacao preparations spread freely to all parts of Europe, though, in 1624 Franciscus Rauch, in a book which had a wide circulation, stated that the consumption of chocolate was largely responsible for the committal of many excesses, especially in the case of monks, for whom he urged that its use should be forbidden.

This, however, was an isolated case of attack against the use of chocolate, and the latter part of the seventeenth century was particularly prolific with its crop of articles and treatises lauding chocolate as a pleasing and nourishing beverage.

The idea that chocolate inflamed the passions was prevalent for a long time, and even as recently as 1712 the *Spectator* states, "I shall also advise my fair readers to be in a particular manner careful how they meddle with romances, chocolate, novels and the like inflammers, which I look upon as very dangerous to be made use of during this great carnival" (the month of May). We have already referred to the probable accuracy of the statement in the *Introduction*.

Chocolate made its first appearance in France in the reign of Louis XIII., probably through the agency of Carletti who was teaching all Europe the process of roasting the beans, in spite of the fact that the Spaniards were still jealously attempting to guard the method of preparation.

It is recorded in a number of places that chocolate was introduced into Europe from Mexico in 1520 and sold in London coffee houses in 1650. It is around the latter date that we find a flood of published references, though, as in modern times, there can be found one original writer from whom many others have drawn their information.

In 1649, there was published "'A New Survey of the West Indies,' by the true and painful endeavours of Thomas Gage, now Preacher of the Word of God at Acris in the County of Kent." In this work were described the origin

of the name "chocolate," the method of preparation, the process of drinking the beverage, and the original story which has already been given. The last-named is described in these words: "But how might this Cacao with the other Indian ingredients be had in England? even by trading in Spain for it, as we doe for other commodities; or not sleighting it so much as we and the Hollanders have often done upon the Indian seas: of whom I have heard the Spaniards say that when we have taken a good prize, a ship laden with Cacao, in anger and wrath we have hurled over-board this good commoditie, not regarding the worth or goodness of it, by calling it in bad Spanish Cagarula de Carnero or sheep's dung in good English."

Gage's work has been freely quoted by most of the early writers, though a book published by one Johannes de Cardenas in Mexico, in 1609, and called "Del Chocolate," seems to have been overlooked. Cardenas states that the drinking of chocolate had become quite common not only in the Indies but among the Italians, Belgians and Spaniards, especially at the court, and that he had not seen anything written upon the subject except by a certain Doctor Marchena. The same author describes the method of making the sweetmeat or drink with crushed cacao beans flavoured with various substances, such as pepper, vanilla, aniseed and sugar, of which last he personally liked a little but not too much. According to Cardenas, the sweetmeat was made up into little balls by the Mexican women and exposed for sale in the taverns.

In 1652, we find a book entitled "Chocolate, the Nut of the Cocoa Tree manufactured in a peculiar manner," but, apart from a few recipes, the work is without interest. It is, however, recorded that just previously, in 1650, coffee and chocolate began to be frequently taken at Oxford.

The *Public Advertiser* (Tuesday, June 16th) stated, in 1657, that cocoa was first introduced into England in Queen's

Head Alley, Bishopsgate, though, with the exception of a repetition of this statement extracted from "*Mercurius Politicus*," in June, 1659, no confirmation can be found.

In the reign of Charles II., a booklet entitled "*India Nectar or a Discourse concerning Chocolate*," announced that chocolate could be bought in East Smithfield for 6s. 8d. per pound, a price which, even taking into consideration the altered value of currency, seems remarkably low. The author of this work, Dr. Henry Stubbe, scholar and physician to the King, has drawn largely from Gage's work, already mentioned, and adds little to our knowledge except to say that: "The northerly tract thereof (of America) principally seems to use the drink chocolate in New Spain, Mexico and the neighbouring Provinces . . . and, indeed, it hath prodigiously spread itself not only over the West Indies, but over Spain, Portugal, Italy, France, High and Low Germany, yea Turkey and Persia; and hath been recommended by sundry learned physicians to the world." Thomas Rugges, in "*Mercurius Politicus Redivivus*," 1659--72, mentioned "There were also att this time a Turkish drink to bee sould in every street, called Coffee and another kind of drink called Chocolate which was very harty drunk."

Even at this time, the best chocolate must still have been a luxury. In 1665, Le Comte de Cominges, the French Ambassador in London, writes in his correspondence, "I wait only till Persod, the King's Messenger, comes back to send to you two cakes of Chocolate, the best in the world, with which I have been presented by the Spanish Ambassador."

That chocolate has not been entirely free from scandal may be deduced from the lines, penned by Andrew Marvell in 1667, after the sudden death of Lady Denham who was supposed to have been poisoned in a cup of chocolate:

"What's frost to fruit, what's arsenic to a rat,  
Was to fair Denham—chocolate."

Uses were, however, quickly found for preparations of cacao by the needy government of William and Mary, which, in order "to raise the rest of the supply for their Majesties resolved to introduce a bill to forbid the sale of beer, ale, cider, mum, coffee, tea and chocolate without a licence." In 1690, on June 2nd, the resolution was carried.

There is an amusing and interesting recipe given by Colmenero, in 1681, which includes the addition of chiles or Mexican pepper, aniseed, powdered roses, logwood, sugar, almonds, nuts and a variety of other flavouring matters to cocoa for those "qui jouissent d'une bonne santé." In spite of the complexity of the decoction, which must have been very expensive to prepare and, incidentally, very nasty and of doubtful virtue, cocoa preparations worked their way speedily into popular favour, for we read in a letter dated February 11th, 1671, that "Madame de Sévigné est desblée de penser que sa fille partie pour Lyon n'y trouvera pas de chocolatière."

In 1659, one Chaillon David obtained the monopoly in France for making and selling chocolate, a privilege which was renewed in 1666 for twenty-six years. His factory is said to have been in Paris near the Croix du Tiroir at the corner of Rue de l'Arbre-Sec and the Rue Saint Honoré.

Under Louis XIV. the use of chocolate became very general in the country, and the advent of the first crop of cacao grown in the French colony of Martinique, in 1679, marked the first step of organised cultivation.

In 1692, the monopoly of manufacture and selling chocolate, which had been previously in the hands of David, was transferred to Damame, who was entitled to hold it for six years. There is, however, on record the advertisement of one Renaud who also sold chocolate in this year and who, after describing in flowery language the delicacy

of the flavours of vanilla and ambergris when added to chocolate, ends thus poetically :—

“ Mais sans vous tant inquieter,  
Pour l'avoir agréable  
Allez chez Renaud l'acheter,  
On l'y trouve admirable.  
C'est chez lui vrai chocolat  
Que se tient la fabrique.  
Le voulez-vous bien délicat  
Ce marchand est l'unique.”

One year later, in 1693, we find the privilege was extended to all confectioners and grocers, Louis XIV. apparently seeing in the new industry a means of increasing his private revenue. This was but following the lead of the English politicians of 1690.

The price of cocoa must have fallen somewhat during this year, for we read that, in 1693, the French King “ mis le chocolat à la disposition de ses invités les jours de réception . . . le chocolat ne coûtait à ce moment que six francs la livre.”

At this time there were only very small quantities of cacao in the hands of private individuals, as the high price of the beans, which were only obtained through Spain from Central America, was a serious hindrance to the general use of chocolate, and it is interesting to note, when we come later to discuss the world's output of cacao at the present time, that the man Rodolphe de Canvillet, whose name should be handed down to posterity for declaring to the revenue officers the greatest amount of cacao in any individual's possession, was the owner of exactly 10 lbs., and that the total amount of beans declared in France did not exceed 22 lbs. This, however, does not indicate that a great deal more cacao was not actually in existence in the country.

In about 1680, the Spaniards had carried cacao from Acapulco to the Philippine Islands where, we are told by



Blanco ("Fl. de Filipinas," 2nd edit., p. 420), it thrived exceedingly, though no record of the arrival of the first Philippine crop is recorded.

Chocolate was freely taken both in England and Germany in the middle of the seventeenth century, when coffee and chocolate houses were in high vogue in both countries. "White's," of the Haymarket, celebrated for its chocolate and fashionable customers alike at the commencement of the eighteenth century, was typical of the many which existed at that time. It may be remembered that White's Chocolate House is represented by Hogarth in the fourth picture of his "Rake's Progress."

Another chocolate house is recorded as existing under the House of Lords about this time, since inside a book was found: "Bought in ye Chocolate House under ye House of Lords, 1712."

The old "Cocoa Tree" coffee house, situated in St. James's Street, Piccadilly, was a Tory *rendezvous* of Queen Anne's time, and was afterwards transformed into a club, similar to "White's" which was in the same street.

Ozinda's Chocolate House, next to St. James's Palace, was another Tory resort, but its owner was arrested in 1775 for conspiracy with the Jacobites.

The "Cocoa Tree" sign survived as late as 1808, but is then recorded as at a shop of a tea dealer in 302, Holborn. Another chocolate house is mentioned as being next to Slaughter's Coffee House in 1742, and yet another at Blackheath.

There were, doubtless, many chocolate houses doing business about this time, though the high prices of the preparation must have kept it from the reach of all but the very wealthy.

It is learned that a silver chocolate mill was a necessary piece of equipment, but the present writer has been unable to find one of authentic antiquity. Probably it was similar

to a coffee mill and was subsequently used for that purpose. One such mill was advertised as being lost at, or stolen from, the "One Bell" in the Strand, and, as it was engraved with "three boars' heads on fess of lozenge," it is presumed that it belonged to one of the titled gentry.

•• A patent was taken out in 1780 by Walter Churchman for making chocolate without fire. It was claimed that chocolate, prepared in this manner, dissolved immediately, was smooth to the palate, of full flavour and left only a very fine sediment, "being by the method made free from the usual Grit and Roughness so much disliked." This is particularly interesting in view of the elaborate machinery required to-day to secure the same end. This chocolate could be sold at 5s. per pound, with "Vanelloes" at 6s. per pound in competition with that sold by the best makers at 6s. per pound and "Vanelloes" at 7s.; but he it observed, "To be sold (only for ready Money) at his Chocolate Warehouse; at Mr. John Young's in St. Paul's Churchyard and by L. Brouse, Haberdasher of Hats in Great Turnstile, Holbourne."

In 1742, in the *Daily Advertiser*, is a reference to E. Bence, widow of Peter Bence, chocolate maker in Broad Street, Soho, who "continues to make and sell all sorts of Chocolate after the Manner of Mr. Alphonse, Confectioner to the late Countess Dowager of Albemarle."

Thereafter, references become much more frequent, a recipe in the *Gentleman's Magazine*, 1791; the formation of a Cocoa Tree Club in London in 1747; a patent by Joseph Storrs Fry, May 7th, 1795, etc.

In 1820, duty was paid in this country on 276,821 lbs., and, three years later, imported chocolate was allowed to enter England. The following thirty years saw many developments in the system of taxation of cacao and cocoa preparations, but, apparently, by this time the sweetmeat had got a hold upon the British public. In 1866, we find

176,959 lbs. of cacao and cocoa preparations imported into England from France. More recent statistics are given in later pages.

Dr. Lindley, Professor of Botany, in a lecture to the Society of Arts in 1851, damned the English chocolate makers with faint praise, saying, "We have the evidence of one of the most skilful brokers in London who has had forty years' experience of the trade, that we never get good cocoa in this country. The consequence is that all the best chocolate is made in Spain and France and the countries where the finer description of cocoa goes. We get here cocoa which is unripe, flinty and bitter, having undergone changes which cause it to bear a very low price on the market." Before the same society, however, in 1874, John Holm said, "The Spaniards did not at first appreciate the virtues of chocolate, and one of their earlier travellers, Hieronymus Benzo, describes it as a 'drink fitter for a pig than a man.'"

It would appear, then, that the taste for chocolate, as prepared in the old days, was "cultivated," for it is seldom that anyone can be met at the present time to condemn it as unfit for human consumption on the ground of unpleasantness.

From an article in *Nature* (II., 497, 1870), written by J. R. Jackson, we gain an insight into the older method of preparation of chocolate. The beans were roasted in a revolving metal cylinder which caused the husks to shrivel slightly, so that they could be removed by fanning. It was stated in the same article that large quantities of the husk were imported from Italy under the name "Miserables," which were used in Ireland by the poorer classes. Jackson warns the people against adulterations of cocoa: "the public, by using them, sacrificing purity for convenience in the preparation for the table." These adulterated powders, he tells us, are prepared by grinding the nibs under heavy

## HISTORY AND GROWTH OF THE CACAO INDUSTRY 15

heated rollers, starch, flour, sugar, molasses and, in the cheaper kinds, other ingredients less wholesome, being added. The names "Homeopathic" and "Soluble" were applied to cocoas of this type, and "Rock" and "Flake" cocoas are also mentioned in this article. Jackson concludes his paper with a regret that the public should have allowed their taste for pure cocoa to have become vitiated, and with a recommendation that "the head of every household might make himself analyst to his own family, and so see that he does not get cheated, either in pocket or health."

Our earliest botanical historians show some divergence of opinion as to the native land of cacao. Humboldt \* states that the cacao tree "grows wild in the forests of the Amazon and Orinoco basins"; Schach † says that it is also to be found in the wild state in Trinidad; and De Cándolle, the greatest authority, doubts whether the cacao plant is indigenous to Guiana, and continues, "Many early writers indicate that it was both wild and cultivated at the time of the discovery of America, from Panama to Campeachy; but from the numerous quotations collected by Sloane ‡ it is to be feared that its wild character was not sufficiently verified."

Townsend's "Dictionary of Dates" records that, in 1649, only one cacao tree existed in the Windward Isles, and that that was grown as a curiosity by an Englishman. In 1655, the cacao tree was found in Martinique, and its cultivation commenced in 1660, but it is not until nearly twenty years later that we find a record of a crop appearing in Europe.

Cacao was said to have been cultivated in Jamaica in the early part of the seventeenth century, but, according to Long in his "History of Jamaica," the plantations were

\* Humboldt, ii., 511.

† Grisebach, in "Flora of British West Indies," 91.

‡ Sloane, "Jamaica," ii., 15

destroyed by a "blast." • It must have been about the same time that disaster overtook the plantations of Trinidad, for Sir A. de Verteuil mentions in his work on Trinidad that, "In the year 1727 a terrible epidemic spread in the cacao plantation," a catastrophe which resulted in complete ruin. Some thirty years later, the cacao industry was revived in the islands by the Capuchin Fathers, who introduced the hardier but inferior Forastero cacao which, Sir A. de Verteuil tells us, is the variety at present cultivated on the island of Trinidad.

Previous to the importation of the Martinique crop into France in 1679, there is little doubt that all the cacao consumed was obtained from Central America, as there is no real evidence to show that the cacao tree was known to be growing wild or in cultivation elsewhere.

The result of such a limited area of supply was, very naturally, to cause cacao preparations to assume a high price in those days, and was, for a long time, a serious hindrance to their general use among all classes. The improvements of means of transport, and the enterprise of planters and manufacturers, have now enabled chocolate in all forms to be placed within the reach of even the very poor, a condition of affairs which has greatly popularised chocolate confections.

The first record of the manufacture of chocolate on the large scale is about 1756, when, it is said, Prince Wilhelm von der Lippe erected a factory at Steinhude and brought over Portuguese workmen especially experienced in the art of chocolate preparation.

France claims to have been the first to erect a factory with mechanical devices for the preparation of chocolate, the honour being due to M. Carbonne, who seems to have been inspired by the possibilities of driving the machinery by water-power in the same way as his oil

*HISTORY AND GROWTH OF THE CACAO INDUSTRY* 17

mill which he conducted in conjunction with his chocolate factory.

In those days, the world's output of cacao was extremely small, and it is interesting to note that, in 1909, it was estimated that a total of 891 million pounds of cacao was put upon the market and that many hundreds of factories were busily at work, in all countries, presenting cocoas and plain and fancy chocolates to an ever-ready public.

## CHAPTER II

### STATISTICS OF THE CACAO INDUSTRY

IN spite of the war, the cacao-producing countries have shown a steady increase in output till, in 1917, the latest year for which figures are at present obtainable, the world's estimated production amounted to over 742 million pounds. Of this, some 320 million pounds were grown in the British Empire.

The increasing supply of, and demand for, cocoa and chocolate are clearly exemplified in the growing imports of the raw materials. England, France, Germany, United States, Holland, Switzerland and Russia have all shown largely increasing imports of cacao beans in pre-war days, the figures for home consumption in each country showing a similarly large increase.

H. Jumelle\* estimated the consumption of cacao in Europe in 1900 as close upon 65 million kilos. Of this quantity † :—

Germany consumed	. 20 million kilos.
France consumed	. 16 „
England consumed	. 12 „
Spain consumed	. 10 „
Russia consumed	. 1 „

This estimation of cacao consumption in England, approximately equal to 26½ million pounds, seems too low when the 1900 official return of raw and manufactured cacao, consumed in the United Kingdom, is compared with it.

\* H. Jumelle, "Le Cacaoyer," 1900.

† The United States, which is not included in Jumelle's estimate, was at the head of all cacao-consuming countries for the year ending June 30th, 1909, a total of 130 million pounds of cacao, raw and manufactured, being recorded for that country.

The following table shows the official return of the consumption of coffee and cacao for the United Kingdom for the ten years 1899—1908 :—

TABLE I.

	Coffee (cwts.).	Cacao (lbs.) (Raw and manufactured).
1899 .	259,949 .	39,972,972
1900 .	260,425 .	44,569,740
1901 .	288,606 .	49,882,354
1902 .	255,567 .	53,688,597
1903 .	268,820 .	51,042,005
1904 .	257,540 .	55,286,457
1905 .	257,612 .	54,872,920
1906 .	255,679 .	52,617,249
1907 .	261,096 .	55,023,420
1908 .	260,675 .	56,566,515

From these figures it will be seen that, whilst the consumption of coffee had remained practically stationary over these years, cacao showed a very steady and pronounced increase.

This increase in favour of cacao over coffee was noticeable in the imports of other countries, notably of Germany, for, according to statements published by the Association of German Chocolate Makers,\* the consumption of coffee increased in that country 24 per cent. between 1886 and 1898, while that of cacao increased 330 per cent. over the same period.

The imports of coffee and cacao for these two extreme years were as follow :—

	Coffee.	Cacao.
1886 .	1,236,305 quintals † .	36,867 quintals.
	(approx. 2½ million cwts.) .	(approx. 8 million lbs.)
1898 .	1,532,704 quintals. .	154,649 quintals.
	(approx. 3 million cwts.) .	(approx. 34 million lbs.)

\* *Verband. deutsch. Schokoladefab.*, xix., No. 7.

† 1 quintal = 1000 cwts.



Unfortunately, comparative figures for later years have not been obtainable.

In Table II. will be found the quantities of raw cacao consumed by the most important countries for the period of the three years prior to the war. •

TABLE II.—*The World's Consumption of Cacao, 1911—1913.*

	1911.	1912.	1913.
	Cwts.	Cwts.	Cwts.
United States . . .	1,160,686	1,303,205	1,330,736
Germany . . .	1,001,031	1,084,293	1,004,927
Holland . . .	463,282	490,545	590,117
United Kingdom . . .	520,568	549,007	564,079
France . . .	638,998	528,193	542,335
Switzerland . . .	193,927	203,572	201,721
Spain . . .	126,804	104,596	121,371
Austria-Hungary . . .	116,140	130,420	121,210
Belgium . . .	108,183	137,630	120,663
Russia . . .	99,207	88,204	103,046
Canada . . .	41,888	59,819	59,045
Italy . . .	43,167	47,872	48,364
Denmark . . .	33,561	33,994	39,801
Norway . . .	20,058	22,164	23,640
Other countries * . .	106,294	118,104	129,914
	4,573,794	4,901,618	5,000,969

An analysis of the statistics for the production of cacao, during the years 1912—1917 inclusive, is shown in Table III., whilst the relative importance of cacao-growing, in some of the chief countries of production, is shown by the following figures :—

*Export of Cacao in cwts. per square mile.*

San Thomé . . . . .	1,487.9
Grenada . . . . .	785.7
Trinidad . . . . .	230.5
San Domingo . . . . .	21.2
Gold Coast (Colony and Dependencies). . . . .	12.6
Ecuador . . . . .	6.6
Venezuela . . . . .	0.8
Brazil . . . . .	0.2

\* Estimated

# STATISTICS OF THE CACAO INDUSTRY

31

TABLE III.—Production of Raw Cacao.

	1912.	1913.	1914.	1915.	1916.	1917.
Gold Coast . . . .	Cwts. 772,983	Cwts. 1,011,071	Cwts. 1,057,764	Cwts. 1,545,560	Cwts. 1,443,336	Cwts. 1,819,280
Trinidad . . . .	370,764	429,610	566,499	482,870	479,393	626,294
Grenada . . . .	101,043	105,264	102,590	120,402	109,772	—
Jamaica . . . .	65,873	46,759	72,290	98,487	64,390	—
St. Lucia . . . .	17,094	14,548	14,232	18,478	14,575	11,866
Dominica . . . .	11,609	9,560	8,602	10,664	5,514	—
St. Vincent . . . .	2,005	1,908	2,010	2,114	1,596	1,191
Montserrat . . . .	33	43	24	29	49	—
Total, British West Indies	568,223	607,349	766,356	703,044	675,259	—
Nigeria . . . .	67,801	72,427	98,777	182,096	179,121	308,841
Ceylon . . . .	71,754	68,526	54,631	83,483	73,245	72,697
British Guiana . . . .	102	505	445	533	416	71
Uganda . . . .	—	—	—	164	258	—
British Honduras . . . .	93	445	184	164	164	—
Fiji . . . .	80	70	108	94	21	—
Mauritius . . . .	—	19	20	20	—	—
Seychelles . . . .	14	15	4	3	—	—
Total, British Empire	1,481,000	1,760,427	1,978,291	2,515,161	2,371,720	2,857,500
Brazil . . . .	690,840	595,180	802,236	885,142	880,347	1,064,561
Ecuador . . . .	708,374	774,723	829,023	728,461	839,606	800,000
San Thomé . . . .	660,571	657,651	655,855	588,271	652,797	697,753
San Domingo . . . .	410,069	383,264	408,385	397,960	414,293	478,000
Venezuela . . . .	281,245	291,402	352,127	359,724	298,760	394,437
Cameroon . . . .	89,580	103,636	80,000	—	—	—
Fernando Po . . . .	43,876	55,588	61,866	76,058	65,909	73,736
Dutch Guiana . . . .	19,006	30,077	37,202	32,611	39,632	37,021
Java . . . .	46,534	41,390	31,112	28,713	28,949	30,600
Haiti . . . .	61,306	34,992	41,837	35,067	38,622	30,364
Cuba . . . .	30,368	27,636	36,228	33,060	29,500	29,500
Belgian Congo . . . .	16,633	17,000	9,503	12,200	15,152	15,310
Guadeloupe . . . .	18,196	17,878	22,126	—	—	—
Samoa . . . .	14,400	16,000	13,000	—	—	—
Martinique . . . .	9,860	10,305	8,835	—	—	—
Costa Rica . . . .	6,081	7,559	6,496	—	—	—
Other foreign countries . .	18,400	19,500	20,000	166,000	176,000	179,000
World's total †	4,534,000	4,844,000	5,394,000	5,857,000	5,829,000	6,628,000

The ports of entry have, since the war, been necessarily somewhat modified, and the following particulars, as those most recently obtainable, have been taken largely from a very able article appearing in the *Bulletin of the Imperial Institute* in 1919.

\* *Bull. Imperial Institute*, 1919, xvii., 1.

† Figure for 1915.

‡ Estimates.

§ Included in "other foreign countries."

|| Colombia, Mexico, Togoland, Gaboon, German New Guinea, Madagascar, French Guiana, German East Africa, Ivory Coast, Dahomey, Réunion and New Caledonia; and in 1915—17, also Cameroons, Guadeloupe, Samoa, Martinique and Costa Rica.

¶ Approximate figures.

*New York.*—Since 1913, America has become the greatest consumer of cacao, the total importations for the year ending June 30th, 1917, amounting to over 150,000 tons, valued at eight and a quarter million pounds sterling. In 1918, they had increased to 180,000 tons, valued at over eight and a half million pounds sterling.

The bulk of the cacao imported into America is consumed in that country, though during 1915 and 1916, there was an exceptionally large re-export trade to Denmark and Sweden, but, in 1917, the re-exports to Europe fell off considerably. The imports from the chief countries into America, during the years ending June 30th, 1914 and 1918, were:—

Imported into America.		1913—14		1917—18.	
From		Cwts	Percentage of total.	Cwts	Percentage of total
British West Africa		70	-	887,474	24.9
Brazil		230,984	14.7	815,639	22.9
Ecuador		234,998	14.9	685,595	19.2
British West Indies		303,414	25.0	459,277	12.9
Dominican Republic		239,134	15.2	355,814	10.0
Venezuela		35,745	2.3	185,978	8.2
United Kingdom		115,211	7.3	9,269	0.24
Portugal		158,385	10.1	1,204	0.03

A simultaneous decrease in the imports of prepared cocoa into America and a corresponding increase in exports of locally-made cocoa products should be noted, thus:—

	Imports (foreign merchandise).		Exports* (domestic merchandise)
	Quantity	Value	Value
	Cwts.	£	£
1912—13	30,988	164,100	78,403
1913—14	27,647	147,123	70,196
1914—15	21,675	121,857	402,951
1915—16	20,957	127,576	347,637
1916—17	16,335	115,237	719,067
1917—18	2,428	19,770	1,365,500

\* Quantity exported not recorded in official trade returns.

The prepared cocoas and chocolates were obtained mainly from the Netherlands, and smaller quantities from the United Kingdom. Up to 1914, some cocoa products were received from both Switzerland and Germany.

*Hamburg.*—Hamburg was, before the war, with the exception of the years 1909 and 1910, the largest European cacao port. The year 1913 showed a net import into Holland alone of 590,000 cwts., valued at £1,634,000. The total imports for that year into Holland were 848,000 cwts., of which nearly 90 per cent. came through other European countries. Of this quantity, Java provided 41,700 cwts., the West Indies 23,100 cwts., and South America 22,500 cwts. of raw cacao as direct importations in 1913. Amsterdam received the greater portion of the Java-grown cacao.

Cacao used to enter Germany through the port of Hamburg before the war. The quantities, consumed in that country, fluctuated somewhat and amounted to some 10,000 or 15,000 tons below the consumption of the United States. It is interesting to record that about one-third of the cacao, imported into Germany in those days, came from British possessions, mainly from West Africa.

*Havre.*—In 1909 and 1910, Havre occupied the premier position as chief port of entry for cacao in Europe. In 1913, it was passed again by Hamburg. France consumed 542,000 cwts. of raw cacao, valued at £1,930,000 in 1913, and, in addition, 58,000 cwts. of cocoa products, valued at £340,000. The cacao entering Havre came chiefly from British West Africa, British West Indies, Brazil, Venezuela, Dominica and Ecuador.

*London.*—In 1914, London held fourth place among the cacao importing ports of the world, Hamburg, Havre and New York, in the order named, coming before it.

“Assuming 1913 to be a normal pre-war year, the British possessions produced in that year about two and a half times as much raw cocoa (cacao) as the United Kingdom

TABLE IV.—Imports of Raw Cacao into the United Kingdom from Chief Producing Countries.

From—	1913.		1914.*		1915.		1916.		1917.	
	Imported.	Retained.	Imported.	Retained.	Imported.	Retained.	Imported.	Retained.	Imported.	Retained.
Gold Coast	166,756 £ 470,937	159,607 447,144	165,185 450,454	145,384 398,780	623,000 2,100,944	393,173 1,244,919	846,558 2,944,697	590,139 915,149	756,730 2,147,562	691,130 1,945,048
British West Indies	18,575 £ 48,047	17,116 43,778	18,575 48,047	17,116 43,778	18,575 48,047	17,116 43,778	18,575 48,047	17,116 43,778	18,575 48,047	17,116 43,778
Ecuador	70,071 £ 284,094	62,506 254,044	70,071 284,094	62,506 254,044	70,071 284,094	62,506 254,044	70,071 284,094	62,506 254,044	70,071 284,094	62,506 254,044
Brazil	118,191 £ 315,143	95,090 254,044	118,191 315,143	95,090 254,044	118,191 315,143	95,090 254,044	118,191 315,143	95,090 254,044	118,191 315,143	95,090 254,044
Nigeria	14,711 £ 41,701	12,659 35,446	14,711 41,701	12,659 35,446	14,711 41,701	12,659 35,446	14,711 41,701	12,659 35,446	14,711 41,701	12,659 35,446
German West Africa	14,087 £ 41,087	12,246 35,446	14,087 41,087	12,246 35,446	14,087 41,087	12,246 35,446	14,087 41,087	12,246 35,446	14,087 41,087	12,246 35,446
Ceylon	164,681 £ 41,087	12,246 35,446	164,681 41,087	12,246 35,446	164,681 41,087	12,246 35,446	164,681 41,087	12,246 35,446	164,681 41,087	12,246 35,446
Venezuela	164,681 £ 41,087	12,246 35,446	164,681 41,087	12,246 35,446	164,681 41,087	12,246 35,446	164,681 41,087	12,246 35,446	164,681 41,087	12,246 35,446
German Pacific Possessions	10,158 £ 27,565	10,158 27,565	10,158 27,565	10,158 27,565	10,158 27,565	10,158 27,565	10,158 27,565	10,158 27,565	10,158 27,565	10,158 27,565
Portuguese West Africa	1,806 £ 11,698	1,806 11,698	1,806 11,698	1,806 11,698	1,806 11,698	1,806 11,698	1,806 11,698	1,806 11,698	1,806 11,698	1,806 11,698
Java	5,570 £ 22,415	1,352 5,669	5,570 22,415	1,352 5,669	5,570 22,415	1,352 5,669	5,570 22,415	1,352 5,669	5,570 22,415	1,352 5,669
Colombia	2,076 £ 5,011	1,871 4,784	2,076 5,011	1,871 4,784	2,076 5,011	1,871 4,784	2,076 5,011	1,871 4,784	2,076 5,011	1,871 4,784
Haiti	1,350 £ 3,375	1,350 3,375	1,350 3,375	1,350 3,375	1,350 3,375	1,350 3,375	1,350 3,375	1,350 3,375	1,350 3,375	1,350 3,375
Spanish West Africa (including Fernando Po)	1,350 £ 3,375	1,350 3,375	1,350 3,375	1,350 3,375	1,350 3,375	1,350 3,375	1,350 3,375	1,350 3,375	1,350 3,375	1,350 3,375
San Domingo	20,180 £ 6,814	19,020 6,282	20,180 6,814	19,020 6,282	20,180 6,814	19,020 6,282	20,180 6,814	19,020 6,282	20,180 6,814	19,020 6,282
Dutch Guiana	1,076 £ 2,756	1,076 2,756	1,076 2,756	1,076 2,756	1,076 2,756	1,076 2,756	1,076 2,756	1,076 2,756	1,076 2,756	1,076 2,756

\* Re-exports greater than consigned imports.

*Imports of Raw Cacao and Cocoa Products into the United Kingdom from Countries not producing Cacao.*

Switzerland: Preparations of cacao, etc.†	94,317	95,924	89,979	394,771	170,070	168,139	140,032	444,640	41,709
Netherlands: Raw cacao	784,668	812,382	767,011	1,725,328	1,500,602	1,672,391	1,447,517	535,732	506,993
Preparations of cacao, etc	5,129	13,060	13,449	•	•	•	•	131	181
Cacao butter	15,721	13,060	84,139	76,483	71,402	49,747	44,496	10,925	10,963
Portugal: Raw cacao	112,402	579,833	554,284	511,782	459,813	371,405	330,186	95,097	91,611
France: Raw cacao	823,450	27,340	29,699	8,437	7,691	18,009	8,711	11,317	3,053
Preparations of cacao, etc.†	17,356	185,082	181,249	63,319	57,349	137,903	77,351	11,324	38,453
Germany: Raw cacao	14,314	113,620	111,346	18,991	16,991	38,314	77,900	12,997	9,817
Preparations of cacao, etc.†	73,527	61,721	61,497	13,418	9,592	30,571	299,464	43,373	29,063
Preparations of cacao, etc.†	33,442	29,788	43,699	41,418	40,290	33,378	27,076	178	174
Germany: Raw cacao	1,897	1,819	2,742	2,603	1,811	1,923	1,897	342	342
Preparations of cacao, etc.†	17,293	16,753	21,972	11,311	10,885	14,365	14,373	4,565	4,565
Preparations of cacao, etc.†	102,131	100,811	49,191	•	•	•	•	•	•
Preparations of cacao, etc.†	27,274	9,156	47,396	•	•	•	•	•	•
Preparations of cacao, etc.†	156,943	70,347	69,410	•	•	•	•	•	•

*Summary of Values (£) of Chief Importations of Raw Cacao and Cocoa Products.*

Raw Cacao:—									
British possessions	1,158,146	886,292	1,204,078	908,187	3,245,568	2,060,491	4,417,389	3,048,064	3,010,442
Foreign countries, producing	814,729	828,321	1,066,563	664,037	2,080,146	1,326,040	1,706,777	1,292,785	1,44,059
" " non-producing	258,043	216,148	166,272	183,270	141,220	101,354	371,969	326,960	43,702
Total	1,040,772	942,660	1,234,835	827,307	2,201,366	1,627,394	2,090,766	1,619,745	497,752
Cocoa preparations (all foreign).	2,108,918	1,731,871	2,428,913	1,735,474	5,748,954	3,606,885	6,498,115	4,666,709	3,498,194
Total cacao and cocoa products	1,948,775	1,891,018	1,660,516	1,262,484	4,220,939	2,937,649	8,216,296	1,960,477	648,733
	4,145,693	3,622,869	4,108,429	3,237,956	8,967,893	5,734,534	6,714,441	6,586,156	4,144,932
									2,628,122

\* Re-exports greater than re-imports.

† Including cacao butter.

TABLE V.—Exports of Raw Cacao and Cocoa Preparations from United Kingdom.

		1913.		1914.		1915.		1916.		1917.	
		Cwts.	£	Cwts.	£	Cwts.	£	Cwts.	£	Cwts.	£
Union of South Africa	Raw cacao	601	2,101	514	1,695	437	1,507	102	3,726	92	—
	Cocoa preparations, United Kingdom	17,454	120,251	14,878	103,171	18,341	132,387	17,282	124,741	24,101	—
	Cocoa preparations, foreign	2,276	20,036	2,482	20,730	4,070	40,749	1,887	17,082	5,820	—
	Total value			125,386		175,243		176,209		30,013	
Australia	Raw cacao	8,793	31,824	6,314	19,137	10,821	44,451	11,202	49,382	1,150	—
	Cocoa preparations, United Kingdom	51,121	327,038	46,016	364,024	47,185	321,654	51,861	414,893	7,854	—
	Cocoa preparations, foreign	522	5,249	1,675	13,580	15,778	122,344	16,137	139,025	949	—
	Total value			306,721		488,769		603,300		69,849	
Canada	Raw cacao	5,684	21,125	4,192	15,347	4,199	16,749	9,651	39,182	3,472	—
	Cocoa preparations, United Kingdom	42,866	244,693	31,062	182,659	18,382	112,878	16,388	113,376	22,573	—
	Cocoa preparations, foreign	52	506	17	211	283	1,465	19	197	—	—
	Total value			198,217		131,192		152,755		166,905	
New Zealand	Raw cacao	1,064	3,743	1,749	4,573	1,409	5,703	806	3,328	68	—
	Cocoa preparations, United Kingdom	10,829	72,421	11,475	77,336	12,850	100,120	14,593	123,002	5,906	—
	Cocoa preparations, foreign	372	3,243	2,457	19,066	8,291	65,000	5,460	47,103	237	—
	Total value			81,407		171,123		173,433		40,477	
British India	Raw cacao	8,669	39,721	7,600	34,661	6,154	48,571	11,872	102,907	1,902	—
	Cocoa preparations, United Kingdom	300	3,622	143	1,845	152	2,402	178	2,376	37	—
	Cocoa preparations, foreign	61,343		56,508		50,973		105,384		19,788	—
	Total value			61,343		50,973		105,384		19,788	
Other British Possessions	Raw cacao	14,643	48,411	12,313	71,712	15,482	98,778	21,102	107,186	5,161	—
	Cocoa preparations, United Kingdom*	939	8,515	980	7,739	1,483	13,167	2,249	21,772	7,297	—
	Cocoa preparations, foreign	96,948		81,506		109,953		188,968		34,874	—
	Total value			96,948		109,953		188,968		34,874	
Russia	Raw cacao	16,836	20,374	21,920	26,877	82,315	388,917	91,510	412,497	38	—
	Cocoa preparations, United Kingdom	362	3,219	781	5,790	2,080	15,878	11,093	115,127	6,633	—
	Cocoa preparations, foreign	10	42	182	1,302	4,161	28,791	8,388	47,716	1,201	—
	Total value			62,535		83,969		603,340		83,275	
Netherlands	Raw cacao	19,690	62,720	108,959	325,900	115,524	445,545	111,424	441,285	9,828	—
	Cocoa preparations, United Kingdom	525	2,040	1,313	6,146	7,549	23,138	9,233	33,138	39,097	—
	Cocoa preparations, foreign	246	869	930	3,255	3,102	9,665	363	1,735	—	—
	Total value			60,520		335,501		462,759		80,097	
United States	Raw cacao	19,191	73,245	7,681	27,008	56,310	215,506	99,448	357,084	50,910	—
	Cocoa preparations, United Kingdom	709	4,574	1,065	6,319	7,660	4,249	313	2,280	51	—
	Cocoa preparations, foreign	—	—	—	—	—	—	—	—	—	—
	Total value			77,810		33,327		219,755		156,286	
Italy	Raw cacao	1,666	5,852	4,115	12,045	16,336	58,251	56,947	202,280	8,409	—
	Cocoa preparations, United Kingdom	104	632	417	2,761	2,904	23,988	3,583	37,874	4,534	—
	Cocoa preparations, foreign	—	—	—	—	—	—	—	—	—	—
	Total value			6,484		14,806		240,134		61,969	

[illegible]

### Summary of Values (£).

	1911	1914	1915	1916	1917.
<b>To British Possessions</b>					
Raw cacao	58,801	40,754	68,052	95,241	18,844
Cocoa preparations, United Kingdom	912,548	835,038	912,747	1,070,120	230,400
" "	44,074	63,131	245,927	228,155	25,106
Total	1,015,423	939,913	1,127,725	1,393,516	391,898
<b>To foreign countries</b>					
Raw cacao	416,132	704,400	2,067,372	1,832,149	293,628
Cocoa preparations, United Kingdom	126,413	105,337	225,062	589,657	416,175
" "	10,980	12,145	92,950	82,121	27,079
Total	553,525	821,882	2,385,384	2,503,927	736,882
Total value of all exports of cocoa, etc	1,568,948	1,761,805	3,513,107	3,702,715	1,128,780

• Channel Islands, Straits Settlements, Ceylon, Hong Kong, British West Indies, *Kypr, etc.*  
• British East Africa, British West Indies, Channel Islands, *Kypr*, British West Africa, Rhodesia, *etc.*  
• Norway, Denmark, Spain, *etc.*  
• Java, Portugal, Canary Islands, Turkey and Crete, China, Japan, Philippine Islands, Porto Rico, Panama, Peru, Chile, Uruguay, *etc.*  
• Norway, Denmark, Portuguese East Africa, Porto Rico, Cuba, Chile, Brazil, Japan, *etc.*



imported from all sources. Again, only about one half of the importations of raw cocoa (cacao) into the United Kingdom came direct from her own colonies and protectorates, in spite of the ample supplies available, the other half being from foreign countries. The apparent uneconomical situation is further emphasised by the fact that foreign non-producing countries manufactured the raw product of British possessions and largely supplied the manufactured article to the United Kingdom."

"During the war the situation altered and a continually increasing amount of raw cocoa (cacao) was obtained from British possessions as the following figures show":—

	Total value of raw cacao imported.		From British Possessions.	
	£		£	Per cent.
1913	2,198,918	.	1,158,146	52·7
1914	2,438,913	.	1,204,078	49·4
1915	5,746,954	.	3,545,888	61·7
1916	6,498,155	.	4,417,389	68·0
1917	3,498,194	.	3,010,442	86·0

This is satisfactory so far as it goes, but it is still more gratifying to learn that not only has the United Kingdom imported more cacao from British possessions during the war, but it has taken and retained a larger proportion of their total output.

Tables IV. and V., taken from the *Bulletin of the Imperial Institute*, showing imports and exports of raw cacao and cocoa preparations in and from the United Kingdom during the years of the war, are, perhaps, of greater interest than a written survey.

It is almost impossible to speak of the future so far as Russia and Germany are concerned. The former country before the war chiefly dealt with re-exports of raw cacao from the United Kingdom. In 1916, nearly one-fifth of our

total re-exports went to Russia, but, in 1917, the abnormal conditions led to an almost complete cessation of this trade. Germany, until 1914, held the chief place as importer of raw cacao from the United Kingdom, but it is not possible at this time to foresee the future for that country.

• To those interested in the study of cocoas and chocolates and in the characteristics of these products shown in various countries, the following particulars may appeal :—

Importing country.	District or country of origin.	
France (1913)	British West Africa	110,000 cwts.
	British West Indies	90,700 „
	Brazil	83,900 „
	Venezuela	69,500 „
	Dominica	46,500 „
	Ecuador	41,300 „
	West Africa (other than British)	32,300 „
Holland (1913)	Java	41,700 „
	West Indies	23,100 „
	South America	22,500 „
Switzerland (1913)	Brazil	49,500 „
	Colombia	27,900 „
	Other South American countries	23,800 „
	Central America	47,800 „
	Africa	46,800 „
Austria-Hungary (1913)	British West Indies	70,400 „
	Brazil	12,700 „
	Vid Germany	10,000—11,000 cwts.
	Switzerland	10,000—11,000 cwts.
Belgium (1913)	80 per cent. of about 130,000 cwts. vid United Kingdom, Portugal, France, Germany and Holland.	
	Belgian Congo	19,500 cwts.
Spain (1913)	Fernando Po	55,600 „
	Ecuador	30,700 „
	Venezuela	30,300 „

The statistics, next given, are interesting as showing the progress in the chocolate industry in Germany, Switzerland and other countries during the war. The German figures given are in marks, and, so far as that country's industry is concerned, they do not include A. G. Stollwerck or the private concerns which, unlike the corporations, are not compelled to give all data and figures concerning their profits, etc.

1 (a). *Fifteen of the largest German cocoa and chocolate corporations.*

	1918.	1917.	1916.	1914.
Gross profits . . . .	17,390,460	19,916,130	28,782,368	23,590,291
Depreciation . . . .	3,625,192	3,128,564	6,032,983	4,041,475
Net profit . . . . .	4,930,341	7,482,431	9,004,499	6,680,830

(b). *Figures in (a) including private concerns, approximately.*

Gross profits . . . .	22,000,000	19,916,130	—	23,590,291
Depreciation . . . .	4,000,000	3,128,564	—	4,041,475
Net profit . . . . .	6,000,000	7,422,431	—	6,680,830

2. *Nestlé and Anglo-Swiss Condensed Milk Company, Vevey, Switzerland.*

Gross profits . . . .	86,230,000	67,030,000	46,498,800	37,600,000
Depreciation . . . .	2,230,000	2,070,000	2,051,000	2,400,000
Net profit . . . . .	30,800,000	20,180,000	16,402,481	14,474,811
Dividends, per cent. .	25	25	25	23½

3. *Peter, Caillier, Kohler, S.A.*

Gross profits . . . .	42,780,214	38,147,701	31,500,460	10,495,785
Depreciation . . . .	1,909,941	1,833,736	1,018,190	811,075
Net profit . . . . .	9,290,835	6,616,818	3,510,916	2,379,737
Dividends, per cent. .	22.0 fr.	22.0 fr.	16.9 fr.	14.3 fr.

4. *A.G. Chocolate Tobler, Berne, Switzerland.*

Gross profits . . . .	2,915,955	2,169,687	1,634,444	958,734
Depreciation . . . .	37,495	98,211	221,946	254,382
Net profit . . . . .	1,418,739	979,966	585,002	298,020
Dividends, per cent .	6.12	6.8	6.6	6.4

A new law, which still obtained in Germany in 1919, had reduced the taxes on sales by  $2\frac{1}{2}$  per cent., but the luxury tax had been increased 25 per cent. Honey cakes, confectionery, package chocolate, cocoa powder, chocolate substitutes and any cocoa preparation came under the luxury tax. A package of chocolate weighing, for example, 100 grms., cost 3 marks 50 pfennigs, to which a luxury tax of one mark was added. The whole industry rose up in arms against this imposition, claiming that cocoa and chocolate were essentials and not luxuries.





## CHAPTER. III

### BOTANY AND NOMENCLATURE

LINNÆUS, in his classical work of 1720, gave to the cacao tree the dignified name of "*Theobroma cacao*," signifying cacao as "Food of the Gods."\* In so doing, he was either actuated by a desire to give to the origin of his favourite beverage an honourable and distinguished title, or else he was influenced by a treatise published in 1684 by Buchat, a French doctor, who described chocolate as an invention more worthy of being called food of the gods than nectar and ambrosia.

It is probable, also, that the Mexican word for chocolate "chocolatl" (supposed to be derived from "choco," "cacava," or "cacana" = the fruit of the tree "quacahault," and "latl" = water), supplied him with the species designation.

The genus *Theobroma* is included among the *Buettneriaceæ*, a tribe of the order *Sterculiaceæ*, of which some twenty species are known to occur in the wild state in Central and South America.

*Theobroma cacao* is the one, of the several species native to the tropical regions extending from Mexico to Brazil, which supplies a large proportion of cacao beans for cocoa- and chocolate-making purposes, and which is chiefly cultivated for its good quality and yield.

Among the most important species of the genus *Theobroma* are *T. bicolor*, *pentagona*, *sylvestris*, *ovatifolia*, and *angustifolia*, all said to be quite distinct from *Theobroma cacao*.

*Theobroma bicolor* or "Tiger cacao," has been used in

\* Greek Θεος = God; βρωμα = food.

Nicaragua for confectionery purposes, but, apparently, pure eating chocolate has not been made from the beans. The pods are hard and woody and show ten ribs, of which every alternate one is considerably more pronounced than its neighbours. The beans, obtained from this variety, are medium in size and, when cut, are quite white in colour. J. Jones,\* Curator of the Botanic Gardens, Dominica, submitted bulk samples of the beans to Messrs. J. S. Fry & Sons, Ltd., Rowntree & Co., and Cadbury Bros., to be made into chocolate, and, from the reports returned by these manufacturers, it would appear that this variety is entirely unsuited for chocolate making and is of practically no commercial value.

On the other hand, the beans of *Theobroma pentagona*, or "Alligator cacao," received most favourable reports from two of these chocolate manufacturers, whilst the third doubted whether the peculiar taste, different from the usual variety, would meet with approval from the English public. *T. pentagona* was brought to Trinidad by Hart in 1893, from Nicaragua, and the shoots were grafted to the Forastero stock then in cultivation in the island. The pods possess five distinct ribs and are thin-shelled. The beans are large and, when cut across, are mostly white. This cacao was thought to be of excellent quality and to contain a higher percentage of fat than *T. cacao*. Messrs. Cadbury Bros. report the beans to be "bold and of good colour" when roasted, but not suitable for making drinking cocoa owing to lack of strength.

The recent publications of Johnson and Hart do not throw any further light on the exploitation of cacaos other than those mentioned. Indeed, the former states "at present only three species are known to yield beans of commercial value; these are *Theobroma cacao* L., *T. pentagona* Bern. and *T. sphaerocarpa* Chev." The former writer

\* J. Jones, *W. I. Bulletin*, 1910, x., 337.

does not mention *T. sphaerocarpa*, but he states of *T. pentagona* Bern. that "it promises to become a favourite with West Indian planters owing to the large size of its beans." It is probable, from experiments that were completed in 1911, that a grafting on to *T. cacao* will result in a very satisfactory cacao.

No one seems to have a good word for *T. bicolor*, called "Tiger cacao" from the rank smell of its seeds. This variety is not even in general use among the natives of Veragua, United States of Colombia, where it is indigenous, though it is said to be used for some purposes by the Indians.

It must be realised that the cacao of the West Indies, as indeed of most countries, consists of a heterogeneous mixture of cross-bred varieties of *T. cacao*. Generally speaking, it is difficult to say where one form begins and another ends. The variety of names given to *Theobroma cacao*, growing in different climates and soils, must not be considered as constituting different "species," but rather "varieties," arising from seed selection, cross-breeding and natural causes.

The botanical description of the genus *Theobroma*, as given in Grisebach's "Flora of the British W. Indies," is as follows :—

"Calyx 5-partite, coloured. Petals 5; limb cucullate, with a terminal; spathulate appendage. Column 10—fid, fertile lobes, bi-antheriferous; anthers bilocular. Style 5—fid. Fruit saccate, 5-celled; cells pulpy, polyspermous, Embryo ex-albuminous; cotyledons fleshy, corrugate. Trees; leaves entire; pedicels fascicled or solitary, lateral."

The cultivated tree, *Theobroma cacao*, grows to a height of 25 feet, though it has been met with as high as 40 feet. In Trinidad, the tree averages about 15 to 20 feet in height, the lateral diameter of the branches being about the same measurement.\*

\* J. H. Hart, "Cacao, etc.," 1911.



The flowers of the cacao tree are very small and, in a manner similar to those of many other tropical trees, are carried on the main stem of the branches and on the trunk, singly or in clusters. The blossoms, though of a bright red-hue, are quite insignificant, and, in due season, they are succeeded by pods of an angular cucumber shape, at first green, but becoming yellow and red on ripening.

The period elapsing between "setting" and "ripening" is about four months.

The fruit pods, hanging away from the leaves and in singularly isolated positions on the main and strongest parts of the tree, present a curious spectacle to agriculturists of temperate climes, though in the tropics it is not an uncommon sight among trees bearing heavy fruit (Plate I.). It is probable that such an arrangement of flower and fruit has been produced from the necessity of securing for small flowers prominent positions for their better detection and fertilisation by insects, and of providing strong supports on which heavy fruit could ripen and provide seed for the perpetuation of its kind.

The trees produce leaves, flowers and fruit throughout every month, and, in the West Indies, the crop is gathered twice in the year, the first time in May or June, the second in November or December.

The fruit, when ripe, is of a rich golden-red colour. Within an outer shell, of about half-an-inch thickness, is contained a sweetish pulp, enclosing twenty to forty almond-shaped seeds which, when fermented, finished and dried, represent the cacao beans of commerce.

Opinion is considerably divided as to how the varieties of cultivated cacao should be classified, but, though in the main all systems are based upon the appearance of the beans, there are certain botanical differences which enable a closer system of classification to be established.

J. H. Hart, in his treatise on "Cacao," makes the following divisions of the varieties cultivated in Trinidad :—

- Class I. : Criollo—fine ; thin-skinned.
  - (1) Variety (a) *Amarillo* (yellow).
  - (2) „ (b) *Colorado* (red).
- Class II. : Forastero—thick-skinned.
  - (3) Variety (a) *Cundeamor verugosa amarillo*.
  - (4) „ (b) *Cundeamor verugosa colorado*.
  - (5) „ (c) Ordinary *amarillo*.
  - (6) „ (d) Ordinary *colorado*.
  - (7) „ (e) *Amelonado amarillo*.
  - (8) „ (f) *Amelonado colorado*.
- Class III. : Calabacillo—small-podded ; thick, smooth-skinned ; flat-beaned.
  - (9) Variety (a) *Amarillo*.
  - (10) „ (b) *Colorado*.

Though other systems of classification divide the varieties into more or fewer classes, among the latter being Sir Daniel Morris's \* method of including Calabacillo under Forastero, all existing cultivated cacaos can be included under one of the three headings given by Hart, with the possible exception of *T. pentagona*.

Criollo cacao trees are not so sturdy and do not produce such regular crops as those of the two latter classes. The shells of the pods are soft and relatively thin. The bean, fresh from a Criollo pod, of either red or yellow variety, is quite rounded and, when cut, shows a white, or nearly white, interior, which is not so bitter or harsh to the palate as that of the Forastero which in turn is less harsh than Calabacillo varieties. The shells of beans of Criollo cacao are thinner than those of either of the other varieties.

From Criollo cacaos are obtained the finest beans for chocolate and cocoa making, those of Java and Ceylon

\* D. Morris "Cacao : How to Grow and How to Cure it."

being characteristic of this class. C. J. J. van Hall says "Venezuela Criollo may be considered as the typical Criollo," and he bears out Preuss' statement that Criollo cacao does not include "Trinitario" or "Carupano," which are Venezuelan names for "Forastero." Besides Java and Ceylon, van Hall mentions Samoa, Madagascar and Nicaragua as also providing cacaos of this variety, and another sub-variety, the "Porcelaine," to be found in Java, is identified as possessing a very thin and easily cut fruit-wall, with a smooth surface and insignificant ridges that give it a strong resemblance to "Amelonado," a Forastero sub-variety. Caracas or Mainland cacao is usually placed in the Criollo class, though some of the lower quality "Caracas" is, undoubtedly, of Forastero origin.

Forastero cacaos, which include some of the Venezuelan and certain strains of Caracas varieties, are distinguished by the pale purple interior of their beans when cut fresh from the pod. The shell of the pod is relatively hard and thick and bears deeper and more pronounced channels, running the length of the pod, than either Criollo or Calabacillo cacaos, thus making the cross-section of the pod star-shaped. Van Hall states that "while the countries where true Criollo is to be found are only few, the Forastero is grown in all the cacao-growing countries, either together with Criollo (Venezuela, Java, Ceylon, Nicaragua, Samoa, Madagascar), or alone in different sub-varieties (Ecuador, San Thomé, Trinidad, Gold Coast, Surinam, Antilles, etc.), and while Criollo is very uniform, having, aside from local types, only two varieties, the true Criollo and the unimportant 'Porcelaine,' Forastero is a collective name for a great number of sub-varieties."

Calabacillo cacaos, which Sir Daniel Morris, the Commissioner of Agriculture for the West Indies, includes under the class-heading of Forastero, are formed into a separate

division by Hart. The Calabacillo is, according to van Hall, the lowest Forastero variety, and that writer agrees with Morris in his classification. The same author states that the planter avoids using Calabacillo seeds, knowing that the quality is very poor and that it ferments slowly. The tree of this variety is the strongest grower and hardiest of all and is chosen by planters for growing on very poor soil and under climatic conditions unsuitable for the finer and more delicate cacaos. The pods of Calabacillo are small, smooth and very much more rounded than those of either Criollo or Forastero cacaos. The beans are flat and have a bitter and astringent taste, characteristics which are typical of beans of inferior quality and low market value.

The reader, interested further in the botany and nomenclature of cacao, cannot do better than consult the two excellent works of van Hall and Hart, to which reference has already been frequently made. Both books are voluminous and detailed and are the works of experts in cultivation. It is considered that sufficient information has been provided to enable the reader to look upon cacao beans, as they come into the market, with more than passing interest, though it is impossible here to give further details for their identification without encroaching on the space to be devoted to the subsequent manufacture of cocoa and chocolate products—the object of the present work.

## CHAPTER IV

### THE AGRICULTURE OF CACAO

#### A. SELECTION OF SITE, SOIL, MANURE, SEED.

THE cacao planter, in common with his brethren, the fruit farmer and the agriculturist of every clime, must be a man of considerable observation and determination,\* for the successful culture of his crop is largely dependent upon an intimate knowledge of the natural conditions existing in the place selected for his plantation and upon the pluck with which he faces the possible destruction of his trees by disease or by the "act of God," more to be feared in tropical than in temperate regions.

Science, which of recent years has been directed more and more to agriculture, has proved a useful asset to the cacao planter, helping both his "observation," by giving reasons for observed phenomena, and his "determination," by putting into his hands weapons with which to combat disease.

In these days of severe competition, it is most necessary that the planter should possess modern scientific knowledge of soil values and such other items of agricultural chemistry as concern the improvement in quantity and quality of his crop.

In this and the following chapter, though it is impossible to deal fully with these questions of much economic importance to the cultivator, chocolate-maker and public alike, an attempt has been made to show how the quality and yield of cacao beans may be influenced by the natural conditions of soil, climate and kind, by the application of

\* Vide "Vallima Letters," by R. L. Stevenson, December 1th, 1891.





scientific methods, to the culture of the cacao tree and by the processes rendering the beans fit for consumption.

The details of the agriculture of cacao have been taken largely from Hart's work, to which reference has already been made, supplemented and brought up-to-date by information supplied by personal friends, engaged in cacao planting, and by the recent researches to be found in current journals issued by the Boards of Agriculture of Ceylon, Trinidad and other cacao-producing countries.

*Selection of Site.*—The best results will be obtained on a good loamy soil of reasonable depth, with a moderate amount of lime and sand present, situated in a district where the seasonal fluctuations of temperature do not exceed the limits of 69° to 89° F.

A deposit of decaying vegetable matter, such as would be found on former forest soil, resulting from the fall of leaves and branches, is, of course, of great value, as a natural dressing of this sort would contain nitrogenous plant foods in a readily assimilable form and would constitute a fine manure for a land where the subsoil did not show, on analysis, a sufficient wealth of plant nourishment.

Good drainage is most essential for the successful cultivation of cacao, as, thereby, the withdrawal of surplus and stagnant waters, both detrimental to cacao plantations, is assured.

The presence of salt (sodium chloride) in a soil is deadly to cacao, so that all situations likely to be affected by tidal waters should be avoided. On the other hand, plantations situated high above the sea-level are not economical, owing to the lower temperature experienced at the higher elevation.

J. H. Hart \* admirably sums up the qualities necessary for a perfect cacao plantation site as follows: "An ideal spot on which to found a cacao plantation is a well-sheltered vale, covered with large trees, protected by mountain spurs

\* J. H. Hart, "Cacao," 1900, 2.



from the prevailing winds, well watered, and yet well drained, with a good depth of alluvial soil, on which rests a thick deposit of decayed vegetable matter, easy of access, and in a district distant from lagoons or marshes, for the sake of the proprietor's health. Such a spot in a climate similar to that of Trinidad could not fail to produce regular crops of the finest quality of cacao." (Plate II.)

*Selection of Soil.*—Close observation of a large number of analyses of soils, suitable and unsuitable for the proper growth of the cacao tree, made by Professor Harrison in the Government laboratories of British Guiana, by Mr. Carmody, Government Analyst of Trinidad, and others, has led to the general conclusion that nitrogen, potassium salts, phosphoric anhydride and lime are absolutely essential to soils for cacao cultivation, whilst excess or deficiency of silica and silicates is detrimental.

The percentages of silica and silicates present are chiefly of importance owing to the effect which sand or silica has upon the mechanical state of the soil. Too great a quantity means too light a soil, with too small a proportion of other useful components; a small percentage of sand and silica indicates a heavy clayey soil. Analyses by the first experimenter show that good soils contain nitrogen from 0.1 to 0.309 per cent.; potassium oxide from 0.118 to 1.072 per cent.; phosphoric anhydride from 0.044 to 0.293 per cent.; lime from 0.356 to 4.981 per cent.

Poor soils contain nitrogen from 0.057 to 0.265 per cent. (usually about 0.1 per cent.); potassium oxide 0.029 to 0.109 per cent.; phosphoric anhydride from 0.002 to 0.157 per cent. (usually about 0.05 per cent.); lime from merest traces to 0.5 per cent. (in one isolated case 4.787 per cent.).

The suggestion that these four components of the soil are of the greatest importance is verified not only by our knowledge of plant foods in general, but also by the analyses

of the cacao tree, shoots, leaves and fruit, made by Marceno who estimated that an acre of twenty-year-old trees would contain 201 lbs. of nitrogen, 95 lbs. of phosphoric anhydride, 251 lbs. of potash, and 400 lbs. of lime.

These figures roughly show the composition of the existing tree and, obviously, do not represent specific amounts of components annually or occasionally extracted from the soil, but they clearly demonstrate the need of the existence of these four components, if good and regular growth is to be made by the trees.

Chemical analysis of a soil alone is of little use to the agriculturist, for it is quite possible for land to possess all the ingredients requisite for the growth of any particular plant and yet to be totally unsuited for its successful culture. The plant nourishment may be in a form unsuitable for root absorption, or, again, the mechanical nature of the soil may prevent the valuable part of the components from being properly assimilated.

It is necessary, therefore, for the intending cultivator of cacao to take into consideration many other conditions of great importance, apart from the chemical composition and mechanical state of the soil, such as natural drainage of the land, situation of the plantation with regard to light, air and winds, taken with which chemical analysis of the soil should be a most useful guide and added factor in determining the suitability of the land for cacao cultivation.

*Selection of Manure.*—When the mechanical condition of the land is unsuitable, or, from chemical analysis, it is found that one or other of the necessary components is lacking, a deficiency which will be evidenced by slow and retarded growth of the tree, production of few leaves, premature falling of the crop, disease or blight, it will be necessary to make use of manures to correct and adjust the prevailing conditions.

It is impossible to teach in writing those manures that should be used and how they should be applied, for there are many schools of thought, each claiming their suggestions of kind and application to be the most efficient.

Of chemical manures there is no end, but, of all manures known, the normal product of the farmyard is the one in which the cultivator places most reliance. The *excreta* from horses contain approximately 25 per cent. of solids, of which 3.5 per cent. are potash and soda, 0.4 per cent. phosphoric anhydride, and 0.6 per cent. nitrogen. Such a manure, thoroughly blended with soil before the seed is planted and applied at the rate of 20 to 30 tons per acre, makes an admirable all-round dressing for cacao. As it is probable that such a quantity of stable manure would not be easily obtainable for a plantation of large acreage, the judicious admixture of nitrate of soda, bone-ash superphosphate, or dried blood, would enable the planter to reduce proportionally the quantity of natural manure.

Nitrate of soda has a tendency to cause a luxuriance of foliage; it also causes plants to grow thicker and to mature earlier. It is most suited to clay soils, but, owing to its solubility in water, it cannot be economically applied before the crop is sown.

Bone-ash superphosphate is also suited to clay soils and is intended to promote early maturity of crops.

Dried blood is most beneficial on light soils and, applied in fine powder, at the rate of thirty to fifty bushels per acre, proves a most valuable manure for cacao trees.

Manures, applied when the trees are in full growth, should be carefully pricked into the soil about the roots, the greatest care being taken not to injure the branching roots in any way, a precaution which cannot be too carefully observed.

The injury to the roots and the wounding of any other part of a growing tree allow access of fungi and wood para-

sites and afford a means of escape to the stimulating juices which convey nourishment to every part of the tree. All injuries should be attended with the greatest care, and a prompt dressing of tar and clay applied to the wounds.

• An interesting list of manures, which have proved successful in cacao culture, is given by Wright,\* from whom the following particulars have been taken :—

Country.	Nature of mixture.
Trinidad	Basic slag, 4 cwts. per acre. Sulphate of potash, 1 cwt. per acre.
• St. Lucia •	(1) Basic slag, 8 cwts. per acre. Sulphate of ammonia, 1 cwt. per acre.
„	(2) Basic slag, 4 cwts. per acre. Stable manure, 8 tons per acre. Sulphate of ammonia, 1 cwt. per acre.
Grenada	(1) Basic slag, 8 cwts. per acre. „ Sulphate of ammonia, 1½ cwts. per acre.
„	(2) Basic slag, 8 cwts. per acre. Sulphate of potash, 1 cwt. per acre.
Dominica	(1) Mulching with grass and leaves.
„	(2) Basic slag, 4 cwts. per acre. Sulphate of potash, 1½ cwts. per acre. Dried blood, 4 cwts. per acre.
Ceylon	(1) Lime and organic matter; the latter either as leaves or cattle manure.
„	(2) Basic slag, 5 cwts. Ammonium sulphate, 200 lbs.
„	(3) Manures rich in readily soluble nitrogen and potash.

Cockran † gives the following blends of artificial manures

\* H. Wright, "Cocoa," 1907, 180.

† M. Cockran, "Report of Analysis of the Cocoa Tree Planters' Association of Ceylon," 1898, 11.

as suitable for application to soils found to be lacking in one or other of the necessary components :—

I.		Per acre:
Superphosphate of lime, 36 per cent. soluble	.	182 lbs.
Sulphate of potash, 50 per cent. potash	.	120 „
Nitrate of soda	.	237 „
(Or sulphate of ammonia, 186)		

II.		
Bone meal	.	300 „
Sulphate of potash	.	120 „
Castor cake	.	400 „

III.		
Thomas's phosphate powder	.	360 „
Sulphate of potash	.	120 „
Castor cake	.	570 „

IV.		
Thomas's phosphate powder	.	360 „
Sulphate of potash	.	120 „
Blood meal	.	300 „

V.		
Fish manure	.	500 „
Bone meal	.	200 „
Sulphate of potash	.	120 „

VI.		
Nitrate of soda	.	119 „
(Or sulphate of ammonia, 93)		
Blood meal	.	100 „
(Or castor cake, 200)		
Bone meal	.	150 „
Sulphate of potash	.	120 „

The work of Dunlop \* and Jones,† in Dominica, on the increased yield per acre of cacao and the subsequent financial advantages of the use of manures, is most thorough. It is

TABLE VI.—Main Series. (Jones, Dominica.) Yield for Seventeen Years, 1900—17.

	Plot 1		Plot 2		Plot 3		Plot 4		Plot 5	
	No manure		Phosphate and potash		Dried blood		Dried blood, phosphate and potash		Mulched with grass and leaves	
	Per plot	Per acre	Per plot	Per acre	Per plot	Per acre	Per plot	Per acre	Per plot	Per acre
1900-2	—	—	—	—	No records	were kept	—	—	—	—
1902-3	—	—	—	—	—	—	—	—	—	—
Wet cacao	750	1,063	3,666	1,281	3,588	1,104	3,807	1,145	3,095	1,300
Cured cacao	—	1,138	1,540	—	1,404	—	1,509	—	—	—
1903-4	—	—	—	—	—	—	—	—	—	—
Wet cacao	548	1,956	808	2,786	970	2,694	738	2,545	962	2,600
Cured cacao	—	822	—	1,170	—	1,131	—	1,069	—	1,092
1904-5	—	—	—	—	—	—	—	—	—	—
Wet cacao	673	2,403	814	2,801	970	2,694	970	3,376	1,279	3,457
Cured cacao	—	1,009	—	1,179	—	1,131	—	1,418	—	1,450
1905-6	—	—	—	—	—	—	—	—	—	—
Wet cacao	748	2,672	761	2,631	1,056	2,933	1,040	3,586	1,510	4,105
Cured cacao	—	1,122	—	1,105	—	1,232	—	1,506	—	1,724
1906-7	—	—	—	—	—	—	—	—	—	—
Wet cacao	730	2,607	887	3,059	972	2,700	1,009	3,479	1,536	4,151
Cured cacao	—	1,095	—	1,285	—	1,134	—	1,461	—	1,743
1907-8	—	—	—	—	—	—	—	—	—	—
Wet cacao	903	3,225	1,160	4,000	1,381	3,836	1,180	4,069	1,773	4,702
Cured cacao	—	1,354	—	1,650	—	1,611	—	1,709	—	2,012
1908-9	—	—	—	—	—	—	—	—	—	—
Wet cacao	978	3,492	1,205	4,155	1,377	3,825	1,444	4,074	1,777	4,803
Cured cacao	—	1,467	—	1,745	—	1,607	—	1,946	—	2,017
1909-10	—	—	—	—	—	—	—	—	—	—
Wet cacao	848	3,029	963	3,321	1,167	3,241	1,267	4,369	1,822	4,924
Cured cacao	—	1,272	—	1,395	—	1,361	—	1,935	—	2,068
1910-11	—	—	—	—	—	—	—	—	—	—
Wet cacao	859	3,067	1,007	3,783	1,289	4,581	1,297	4,473	1,890	5,107
Cured cacao	—	1,288	—	1,580	—	1,504	—	1,879	—	2,145
1911-12	—	—	—	—	—	—	—	—	—	—
Wet cacao	804	2,871	1,012	3,490	1,272	3,533	1,272	4,387	1,721	4,651
Cured cacao	—	1,206	—	1,466	—	1,484	—	1,842	—	1,953
1912-13	—	—	—	—	—	—	—	—	—	—
Wet cacao	802	3,196	1,088	3,752	1,512	4,200	1,314	4,531	2,001	5,408
Cured cacao	—	1,338	—	1,576	—	1,764	—	1,903	—	2,271
1913-14	—	—	—	—	—	—	—	—	—	—
Wet cacao	678	2,422	885	3,050	1,133	3,147	1,104	3,807	1,509	4,078
Cured cacao	—	1,017	—	1,281	—	1,322	—	1,599	—	1,713
1914-15	—	—	—	—	—	—	—	—	—	—
Wet cacao	779	2,782	1,049	3,617	1,366	3,704	1,133	3,907	1,715	4,635
Cured cacao	—	1,168	—	1,519	—	1,593	—	1,641	—	1,947
1915-16	—	—	—	—	—	—	—	—	—	—
Wet cacao	715	2,554	1,110	3,827	1,596	3,607	1,069	3,686	1,474	3,964
Cured cacao	—	1,073	—	1,607	—	1,514	—	1,548	—	1,673
1916-17	—	—	—	—	—	—	—	—	—	—
Wet cacao	841	3,361	1,285	4,431	1,497	4,158	1,486	5,124	1,772	4,799
Cured cacao	—	1,412	—	1,861	—	1,746	—	2,152	—	2,011

\* W. R. Dunlop, *West Indian Bulletin*, xvi., 2, 121 et seq.

† J. Jones, *West Indian Bulletin*, xvi., 4, 324 et seq. Also *Annual Report*, 1916—17, Agricultural Department, Dominica.

not intended to give full details of the experiments made, but, owing to the huge increase in production brought about by the use of suitable manures, and, in view of the fact that the present work may reach the hands of those interested in this subject of such great economic importance, Tables VI. and VII. have been included here as most striking examples of the application of science to agriculture.

TABLE VII.—(*Jones, Dominica.*) *Average Annual Return for Fifteen Years.*

No.	Manurial treatment.	Wet cacao.		Cured cacao	Increase over no-manure plot.
		Per plot.	Per acre.	Per acre.	Per cent.
		lbs.	lbs.	lbs.	lbs.
1	No manure . . . . .	790	2,821	1,185	—
2	Phosphate and potash . . . . .	1,013	3,493	1,467	23.8
3	Dried blood . . . . .	1,236	3,433	1,442	21.7
4	Dried blood, phosphate and potash . . . . .	1,156	3,986	1,674	41.3
5	Mulched with grass and leaves . . . . .	1,590	4,297	1,805	52.3
6	Mulched with grass and leaves . . . . .	1,250	5,000	2,100	77.2
7	Cotton-seed meal . . . . .	1,050	4,200	1,764	48.8
8	No manure . . . . .	920	2,222	933	—
9	Mulched with grass and leaves . . . . .	1,515	4,062	1,706	82.6

The above table gives the average annual return of wet and dried cacao, in pounds, for fifteen years in the case of the first five plots, and for nine years in the case of plots six to nine. The last column shows the percentage increase of each plot over that without manurial treatment.

*Selection of Seed.*—Having briefly dealt with the sites, soils and manures most suited to cacao plantations, there remains the very important consideration of seed selection.

Cacao seeds possess but little vitality and readily succumb to dry air and to alternations of periods of damp and dry weather, and they are, therefore, not easily sent to distant parts so as to be in a fit condition for germination at the end of the journey.

The planter should choose large seeds from ripe pods of trees which regularly produce the finest quality of beans. By these means, if the trees do not grow too close to others of inferior quality to run the risk of cross-fertilisation, the planter is more assured of a good, even, all-round crop.

The selection of the variety, Criollo, Forastero, or Calabacillo, must depend upon the soil and climatic conditions prevailing at the place chosen for the plantation.

From Criollo cacao are obtained the finest beans for flavour, but the tree is not so hardy as the inferior-beaned Calabacillo, which produces, also, a larger crop. If the soil of the plantation is poor, Calabacillo cacao will thrive the best, while the Forastero variety, occupying the intermediate position, will grow well in soils and climates where any cacao may be reasonably expected to flourish. Hart has recommended the grafting of the finer quality Criollo to the hardier Calabacillo cacao, thereby obtaining the advantages of both varieties, and the additional benefit of being sure of securing a continual and even crop of the variety selected, which, by reason of possible cross-fertilisation of the flowers of the mother tree, is denied to trees grown from seed.

As in the last chapter, the works of van Hall and Hart are recommended to the reader interested in the agriculture of cacao. Both van Hall and Hart have had considerable experience in cacao cultivation, the former being now Director of the Institute for Plant Diseases and Culture in Java, whilst the latter, for many years in Trinidad, died in 1911 when holding the position of Superintendent of the Royal Botanical Gardens, Trinidad.



## CHAPTER V

### THE AGRICULTURE OF CACAO

#### B. PLANTING, DISEASES, PRUNING, PICKING.

##### **Planting.**

THE seed selected and the land prepared, it remains for the planter to decide whether he will place the seed direct into the land, when the position which the tree will take up will be permanent, or rear the seedlings in a nursery, till their size and strength fit them to be transplanted to the open plantation.

In the former case, the seeds are carefully placed into the loosely broken ground about 1 inch deep and 6 inches apart, in the form of a triangle, and a little light earth is sprinkled over them and is gently trodden down with the foot. Each triangle is placed about 12 feet apart.

As the trees grow, the weaklings are weeded out, making more room for the stronger-growing shoot to develop. It is a general rule that the higher the elevation of the plantation and the poorer the soil the nearer may the trees be allowed to grow, a distance ranging from 12 to 15 feet each way probably meeting all requirements.

It must be realised that no definite rule can be laid down for planting or for any other of the operations of the planter, which are necessarily only to be learnt by experience, but, for a mental picture of a plantation, the descriptions here given will serve.

In planting in nurseries, the same system of sowing is employed, but added facility for individual and personal





inspection is afforded. In the nursery, the seedlings are tended until some 2 feet in height, care being taken that no direct heat from the sun shall fall on the plants, a danger which is prevented, both in the nursery and in the open plantation, by shelters, either of an artificial nature or of trees which afford plenty of shade.

In Grenada and other hilly districts, the formation of the land is used to advantage for providing the necessary protection from the sun and wind, and in these places, no trees are grown, nor artificial structures erected, for that purpose.

In the lower lands of Trinidad, open to the unchecked influence of the elements, protections from both wind and sun are erected and grown, shelters from the former being usually built outside the plantation, whilst shady trees are grown in rows side by side with the cacao. That protection from the sun is considered not only desirable but essential for the successful culture of cacao finds further proof in the fact that the principal and larger trees selected are usually those providing the greatest shade and are seldom, if ever, of economic value.

Varieties of *Erythrina*s known as "Bois Immortel," or "Madre de Cacao" ("Mother of Cocoa"), to use the Spanish name, leguminous plants of dense shade (Plate III.), are most generally employed in Trinidad, and, though from the nature of the plant they probably aid in accumulating nitrogen in the soil around the roots, their timber is quite useless, even for fuel, and the boughs of the tree have the unfortunate characteristic of falling in wet and windy weather, with probable injury to the cacao growing beneath.

Cacao has been successfully cultivated in Grenada without shade, and the planters consider shade-trees both unnecessary and harmful. Some planters of Jamaica are of the same opinion, and van Hall has stated that in Surinam, though the rainy season is followed by very dry months, cacao could be grown successfully without shade-trees, if

the plantation were properly conducted. Opinion differs on this matter, though the Trinidad planters speak of the necessity of shade-trees in no uncertain voice. Permanent shade-trees for cacao are generally considered necessary in Central America, Ceylon, Trinidad, Fiji, Tobago, British Honduras, Samoa and the whole of West Africa, including San Thomé.

There are many other trees besides those mentioned, grown for the purpose of providing shade and especially favoured in different districts, among the smaller plants being *Zea mays*, castor oil, cassava and banana or plantain, each of which provides seed, fruit or root of some commercial importance.

It is noticeable, however, that, until the rubber plant came into such prominence, the larger trees used for shading cacao were of little or no economic value. The Heveas, from which Para rubber is obtained, are now largely used for shade and, though comparatively slow growing, yield a produce of equal, if not greater, value than the cacao trees they shelter.

Appended is a list of shade-trees commonly used in cacao-producing countries \* :—*Adenanthera pavonina*, *Albizia moluccana*, *A. Lebbek*, *A. odoratissima*, *A. stipulata*, *Erythrina lithosperma*, *E. umbrosa*, *E. indica*, *E. ovalifolia*, *E. velutina*, *Artocarpus integrifolia*, *A. nobilis*, *Cassia Siamea*, *Casuarina equisetifolia*, *Eugenia Jambos* (Jambo), *Filicium decipiens*, *Melia dubia*, *Mesua ferrea*, *Pithecolobium Saman*, *Castilloa elastica*, *Manihot Glaziovii*, *Hevea brasiliensis*, *Gliricidia maculata*, *Pterocarpus indicus*, *P. marsupium*, *Valeria acuminata*, *Acacia pycnantha*, *Cedrela serrata*, *C. Toona*, *Eucalyptus leucoxylon*, *E. marginata*, *Grevillea robusta*, *Michelia Champaca*, *Myristica laurifolia*, *Azadirachta indica*, *Peltophorum ferrugineum*, *Swietenia macrophylla*, *Tamarindus indica*, *Thespesia populnea*, and others.

\* Vide also H. Wright in "Cocoa," 1907; C. J. J. van Hall in "Cocoa," 1914; J. Hinchley Hart, "Cacao, etc.," 1911; W. J. Johnson, "Cocoa, etc.," 1912.

Other plants, which, whilst providing a certain amount of shade, are grown for their commercial value as inter- or catch-crops between the cacao trees, are lemon grass, citronella, cotton, ground-nuts, cassava, chillies, pepper, bananas, yams, coffee, tobacco, rubber, tea, camphor and coconuts.

### Diseases.

A matter of no small importance to be considered in selecting shade-trees is their immunity from disease.

As a general rule it may be stated that the cacao tree is not subject to the attack of parasitic fungi, but ill-drainage of the land or close proximity to other trees affected with fungoid disease may speedily bring about an epidemic in the plantation.

"Canker" and "Black rot" are the two most deadly diseases to which cacao is prone. The former, probably the most widely spread cacao disease, is due to a fungus allied to *Nectria*, which attacks the bark of cacao and other trees, and which has now been named *Nectria Bainii*. This disease has been observed in Antilles, Surinam, Kamerun, Ceylon and Java. The investigations of Rorer,\* in Trinidad, and of Petch,† in Ceylon, have shown that "canker" is due to *Phytophthora Faberi*, which is also often given as the cause of the "black rot" of cacao pods. Experts again differ on this question of identification, the main difficulty arising from the fact that, though it would appear that both diseases, the one to the stem and branches and the other to the pods, are due to the same parasite, they do not necessarily occur side by side on the cacao fields. Indeed, fields that are disposed to an annual outbreak of "canker" often show little or no blackening of the pods, and *vice versa*. Van Hall

\* J. B. Rorer, "Pod-rot, Canker and Chapon-wilt of Cacao," *Bulletin Department Agriculture, Trinidad*, July, 1910.

† Petch, "Cacao and Hevea Canker," *Circ. Roy. Bot. Gardens, Ceylon*, 1910, v., 13.

denies that *Nectria* is the cause of "canker," but admits that the disease is always followed closely by the appearance of this second parasitic fungus, which, however, he does not consider as playing a rôle of any importance. The latter also is due to a parasitic fungus named *Phytophthora omnivora* (or *Faberi*), similar in many ways to the "black rot" of the potato, *Phytophthora infestans* and to *Peronospora* generally.

"Black rot" attacks the cacao pods, sending its filaments well into the interior of the pod which, in time, is turned into a black pulpy mass, whilst the fungus appears on the outside surface of the shell as a white mould. This fungus has been identified in cacao plantations in all districts, especially in Ceylon and Trinidad. There is little doubt that it was *Phytophthora omnivora* which destroyed the crops and prosperity of Trinidad in 1727, a disastrous episode already mentioned as being reported in De Verteuil's book on Trinidad.

A new disease appearing in the West Indian cacao plantations was noted by Bancroft\* in 1910. This parasitic fungus belonged to the genus *Colletotrichum* and had previously been recorded from other cacao-producing centres, especially from Surinam, though this seems to have been its first appearance in the West Indies. It has been mentioned by Hall and Drost,† Delacroix,‡ van Hall§ and Von Faber|| in their studies on fungi parasitic to plants, and, besides being extremely contagious, it causes complete deterioration of the pods, making them hard and woody, and sapping the juices which protect and nourish the beans.

An interesting article on cacao diseases on the Gold

\* C. K. Bancroft, *W. I. Bulletin*, 1910, xi., 34.

† Hall and Drost, *Recueil des Trav. Bot. Néerl.*, 1908, iv., 343.

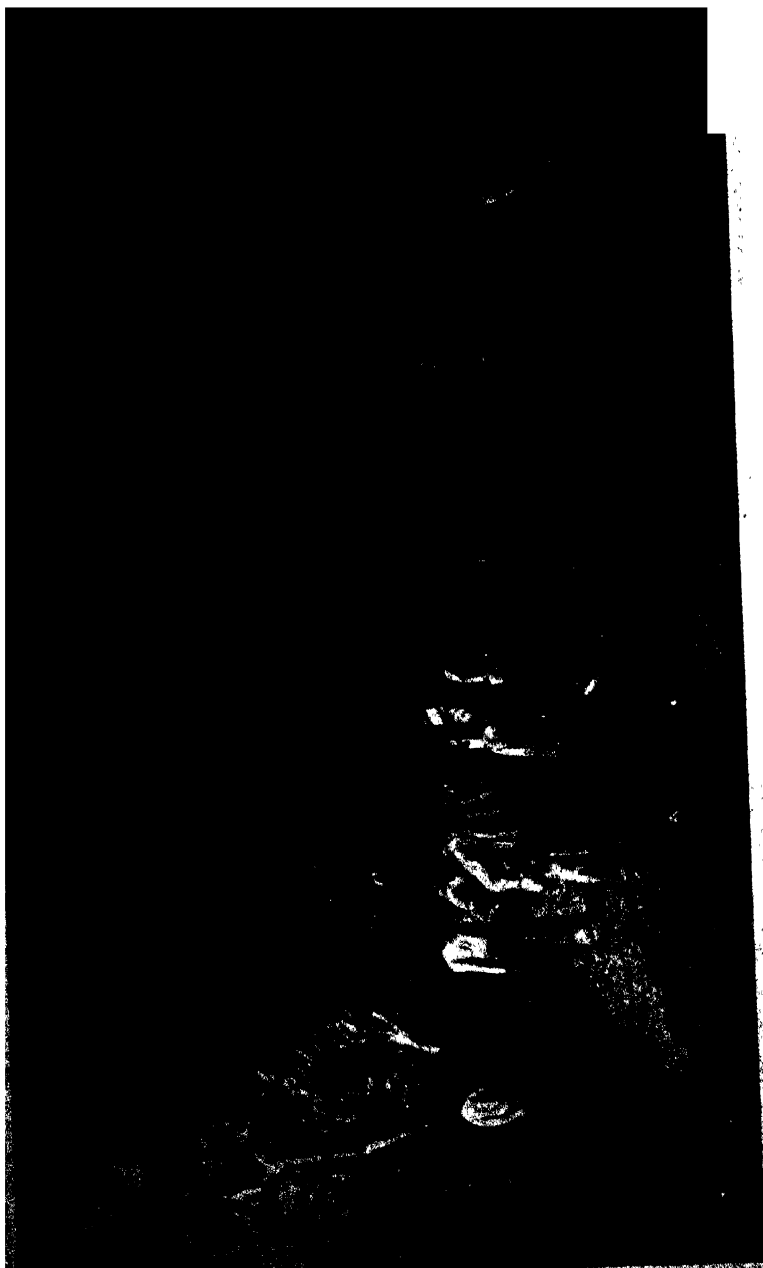
‡ Delacroix, "Champignons parasites des plantes cultivées dans les régions chaudes," *Bulletin de la Soc. Myc. de France*, 1905, xxi., 191.

§ C. J. J. van Hall, "Cocoa, etc.," 1914.

|| F. C. Von Faber, "Die Krankheiten und Parasiten des Kakaobaumes," 1908.







PICKING THE CACAO PODS, TRINIDAD. (See p. 58.)

Coast appeared in *Tropical Life* (July, 1915). It recorded a new disease from Calabar and in the Western Province at Olomu. The first indications took the form of "scaling" from the outer bark in the vicinity of the forks where branches came off. On the branches above and below a fork the bark mostly scaled away, leaving only small patches. No cacao disease of similar symptoms had, at that time, been recorded from Kew.

In the Gold Coast, pod diseases are most common, and many root diseases have also been reported, the latter being due chiefly to *Hymenochaete noxia* (Berk.).

*Thyridaria tarda* (Bancroft), described as an "ubiquitous saprophyte," has caused considerable trouble among the cacao tree roots, though van Hall has doubted whether the so-called "Thyridaria root rot" can be attributed to this fungus.

The suggested remedies are such as would be applied to any tree suffering from disease, namely, the strengthening of plant-growth by addition of manure to the soil, installation of a proper drainage system and the removal of all possible means of contamination, by lopping off the diseased branches of affected trees, and a complete destruction, by fire, of the refuse thereby created.

It is not an uncommon practice for the pods to be shelled on the plantation itself for the removal of the beans, the husks being allowed to rot on the surface of the land until sufficiently decayed to be dug in. This practice constitutes a danger of spreading disease among the cacao trees, and it is most essential that all affected husks should be removed outside the plantation and burnt forthwith. A check on disease may often be brought about by the use of sprays of solutions containing sulphate of iron or copper, fungicides which have proved most efficacious in combating the potato "black rot" and "corn smut" (*Ustilago carbo*) in this country.

Among the insect pests mentioned by Hart as causing the greatest trouble to cacao planters are the "parasol ants" (*Ecodoma cephalotes*) and the "cacao beetles" (*Steirastoma histrionica*).

The "ants," which remove portions of the cacao leaf often afflicted by the growth of certain fungi, upon the conidia of which the ants feed, are most persistent in their depredations, and, in spite of sulphur or tobacco fumes, they will often recur at the sites of their former nests. So much damage has been done in the past to plantations by the ravages of these "ants" that the Legislative Council of Trinidad can pronounce any district, troubled with the pests, to be in quarantine.

The "cacao beetles" deposit their eggs in the bark and wounds of the tree and, by boring small holes through the branches, cause boughs to fall in windy weather. The most satisfactory cure for this pest seems to be the cutting away and burning of infected parts, or the more cautious probing to remove the beetle. In either case, the wounds in the tree must be dressed with tar and clay or other antiseptic mixtures.

The "cacao beetle" *Steirastoma depressum* is more frequently mentioned by agriculturists than *S. histrionica*. A full description of this pest has been made by Ballou, entomologist on the staff of the Imperial Department for the West Indies, and is reproduced in an article which appeared in the *Journal of the Royal Society of Arts* (August, 1909, 793). *S. depressum* has been reported commonly in the West Indies and Surinam, where both the adult beetle and the larva cause immense damage. The adult feeds largely on the tender bark of the young plants and deposits its eggs in crevices in the bark. The egg hatches, and the larva burrows into the bark where it feeds, grows and develops. The direct damage done by the larva results in the weakening of the tree, caused by the sap-flow being cut

off, whilst the nibbling of the adult seems to cause more an offence to the eye than any direct damage. The indirect result of the depredations of both stages of the beetle is to cause the tree to be more liable to attack by fungi which, as we have seen, are formidable foes of the cacao planter. Van Hall refers to the beetle as one of the worst pests in Trinidad, Grenada, Guadeloupe, Martinique, Venezuela, Surinam and Demerara, and mentions a paper by Guppy, which we have not consulted, as giving a clear exposition of the life-history of this pest and of the methods of combating it.

The aphid fly and lice, common to all vegetation, are to be found on the cacao tree, and, though no real harm is caused by their presence, the cleanly planter will endeavour to keep them away by spraying and other preventive measures.

Vegetable parasites and orchids, words used indiscriminately in Trinidad for one and the same thing, are many and have been described in articles submitted to the Association of German Chocolate Makers. There is little doubt that all such cancerous growths sap the energy of the tree and should be removed at the earliest opportunity by cutting as close as possible to the point of juncture.

### Pruning.

Parasites and the seeds of disease fall upon the tree under most favourable conditions for their growth when the sap is down, and when the dead wood abounds. It is a mistake, however, to cut away all the old wood at once, as heavy pruning always stays the growth of a tree by reason of leakage of sap which must occur when incisions are made.

The object of pruning is primarily to produce an even, symmetrical tree with plenty of foliage and fruit, to remove boughs which are past bearing and to induce new growth.

In young trees with a single shoot, three or, at the most,

four small branches should be allowed to form at the sides with as much regularity as circumstances will permit. In the same way, by judicious pruning, the secondary and tertiary branches should be induced to grow at regular intervals.

In the early stages, the wood should be pruned when in a state to be "pinched" and not cut, and, whenever a knife is employed, the greatest care should be taken that ragged cuts are not made.

Later, when knife and saw are brought into use, symmetry, by removal of the oldest wood and encouragement of young shoots in the most desirable positions should be aimed at, and the wounds should be covered with some antiseptic mixture.

Pruning is best carried out at the close of a crop, when both flower and fruit are absent, and, though it is often desirable and necessary to prune whilst the crop is on the trees, care must be exercised lest injury occur to the fruit and, more essential still, to the flower of the next season's fruiting.

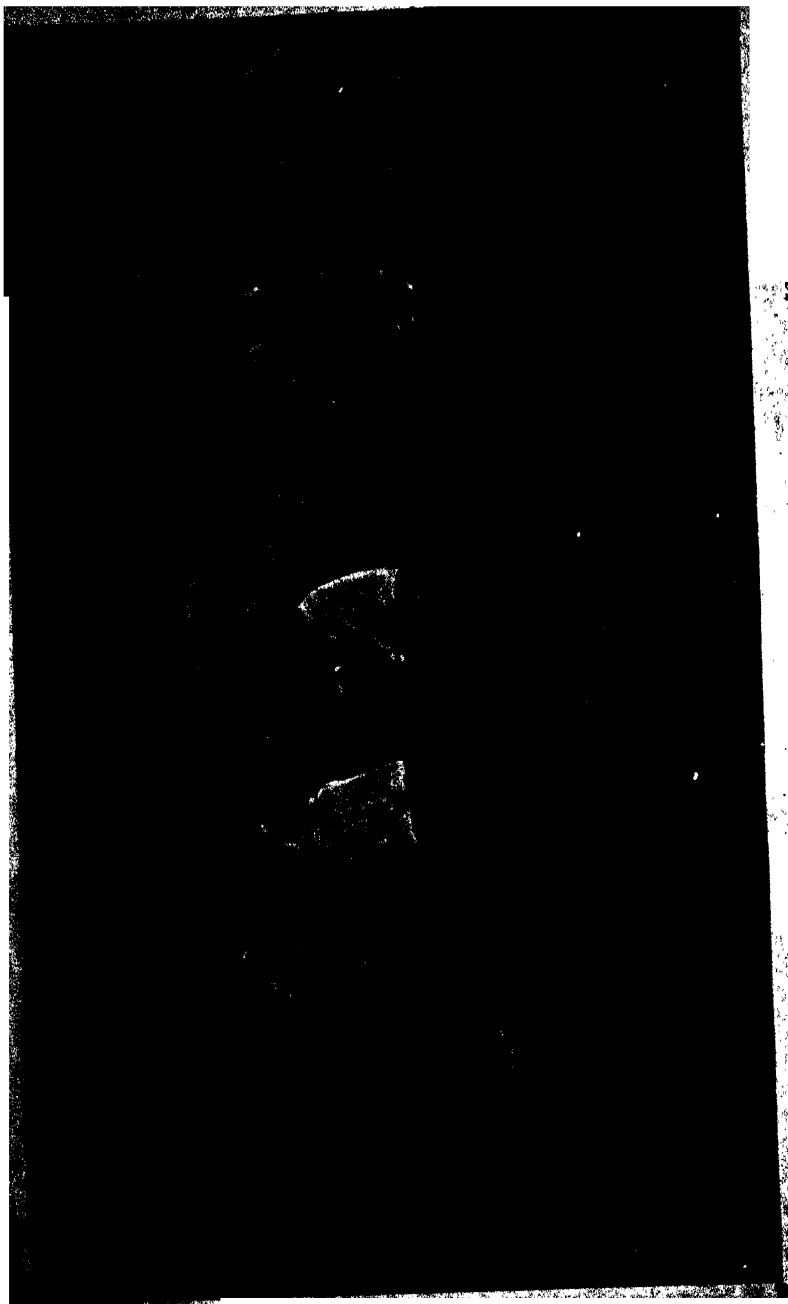
### Picking.

When the trees are three or four years old they commence to flower, and, after they have once produced fruit, regular crops may be expected for fifty or more years, if proper care is taken. The yield usually continues to increase a little till it reaches its maximum in the tenth, eleventh or twelfth year.

The fruit, which has already been described, is gathered, when in reach, by means of a hand knife, the cacao hook or pruning knife, fitted to the end of a long stick or bamboo, being used for gathering the fruit on the higher branches. (Plates IV., V. and VI.)

The flowers for the following crop usually occur at the point of juncture of the fruit with the tree; consequently,





CARRYING THE CACAO PODS TO THE SLICING HEAPS, TRINIDAD. (See p. 50)

the picking or cutting is attended by considerable risk of injury to the blossoms, and it is important that the cut should not be made too near to the main support.

Colour and sound on tapping are the most sure guides to the planter as to whether the pod is ripe for picking, but it is admitted on all sides that great experience must be acquired before absolute certainty as to the ripeness of a pod can be ascertained by these means. For this reason, picked men of experience should be selected for gathering the fruit, as the beans of unripe or over-ripe pods, if not discarded, will lower the standard of the bulk.

If only ripe pods are gathered, the sorting which they must undergo is greatly simplified, and the planter has the satisfaction of obtaining his cacao in the finest condition.



## CHAPTER VI

### FERMENTATION OF THE BEANS

FERMENTATION, which has been looked upon from very early times as a necessary process for obtaining cacao of finest aroma and flavour, may be regarded primarily as the most convenient method of removing adhering pulp and of obtaining the bean in a suitable state for quick drying.

It is undoubtedly true that the cacao commanding the highest market price is usually fermented, so that a good deal may be said for the first argument in its favour. However that may be, the difficulty of removing the pulp clinging to the beans and of rapidly drying the cleaned seeds, without allowing germination to commence, would be accentuated, if fermentation were not resorted to.

Germination of the bean would entail a loss of a certain amount of fat, but, by quickly killing the embryo and life of the seed by fermentation or sweating before the more readily assimilated sugars are used up, this valuable portion of the bean is preserved.

The value of fermentation has been discussed by many writers, but probably the most complete attempt to reach a conclusion is that made by H. H. Smith, the editor of *Tropical Life*, in his work "The Fermentation of Cacao," published in 1918. In this treatise can be found essays by Dr. Axel Preyer, Dr. Oscar Loew, Dr. Fickendey, Dr. A. Schulte im Hofe, Dr. J. Sack, Dr. Lucius Nichols and others. The book includes not only much original research on cacao fermentation, but the considered judgment of men whose business in life has been the study of the cacao industry.

A great deal of information may be drawn from this work, and the chocolate manufacturer who studies his products cannot do better than obtain a copy of this admirable collection of essays and read it.

Still more recently, Knapp \* has shown a careful comparison between fermented and unfermented beans in Trinidad, and, taking into consideration the fact that this writer is also familiar with the technical side of the cocoa and chocolate industry, his opinion carries no little weight. It is, therefore, intended to examine the differences existing between fermented and unfermented beans of the same kind, as seen by Knapp, and later to study the processes operative in fermentation.

*Comparison of Fermented Cacao Beans with those Dried direct in the Sun.*

" A "

Dried Beans		Fermented Beans.
Shape . . . . .	Flat.	Plumper or rounder.
Shell . . . . .	Soft.	Crisp, usually darker outside ; note the dark brown colouring deposited inside of shell.
Looseness of shell.	Shell tight.	Shell more or less free.
Colour of interior .	Slate colour to black.	Brown ; light and dark browns (and purples).
Consistency of interior	Leather to cheese.	Brittle ; crumbles readily.

" B "

Pod.	Beans.	Average weight per bean in grms.	Number per pound.	Shell per cent.	Moisture in shelled beans per cent.	Butter in shelled beans per cent.
Trinidad Criollo	Unfermented	1.682	270	17.0	5.02	60.22
	Fermented	1.716	260	13.3	4.87	54.74
Trinidad Calabacillo	Unfermented	0.893	510	16.1	5.03	53.33
	Fermented	0.854	535	15.1	5.15	50.01

The above tables show the extremes in differences, and, in practice, the dried beans are usually slightly fermented.

\* A. W. Knapp, " Practice of Cacao Fermentation," from *Tropical Life*, 1919, x., 42.

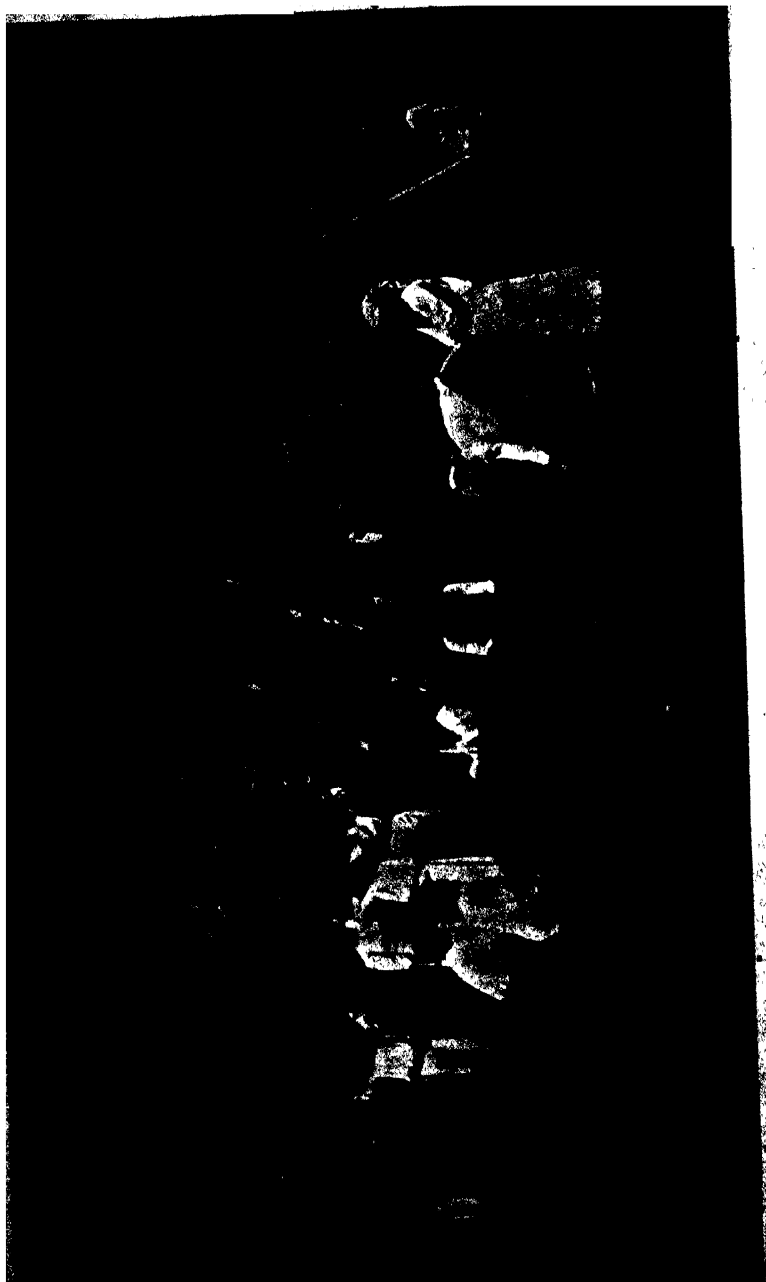
When roasted, however, the product from the unfermented is inferior in every way to that from the fermented beans. This bears out our previous contention and is a matter of common knowledge at the present time.

As we have pointed out often enough, the advantages to the manufacturer of finished products, such as chocolate, biscuits, etc., of a raw material prepared in a certain manner, need not find favour in the eyes of the producer of that raw material. Indeed, the direct advantages to the two producers may be in opposition, though the indirect advantages of placing on the market a suitable raw material can only result in good business to both. Such a case can be seen in this instance where the planter may reasonably object to a loss in weight of his bagged beans, by fermentation, for the advantage of the manufacturer of cocoa and chocolate. Yet it would be a short-sighted policy for the planter to put this forward as an argument for not fermenting his beans. Whether or no this argument carries any weight with cacao planters, whose practice it is to omit fermentation altogether or to conduct fermentation in a slipshod fashion, it is difficult to say. We can give them the benefit of the doubt and quote on their behalf the work of Perrot,\* who recommends the treatment of cacao beans with a 1 per cent. solution of sodium or potassium carbonate and the removal of the pulp mechanically, after the beans have been subjected to heat in the presence of moisture. The same author recommends the sterilisation of plantation cacao, by means of steam, and a subsequent drying, in order to obtain a more uniform product from one and the same plantation. By these means, Perrot claims to obtain a better product than by fermentation. Knapp, at the Third International Congress of Tropical Agriculture,† discussed the value of Perrot's method which had been tried, at the request of the former,

\* E. Perrot, *Compt. Rend.*, 1913, clvi., 1394—6.

† A. W. Knapp, *Tropical Life*, vol. ii., 227 et seq.





A HEAP OF CACAO PODS READY FOR SLICING, TRINIDAD. (See p. 56.)

in Trinidad. He said, "the alkalisèd beans were clean and light in colour, showing that the alkaline carbonate had facilitated the removal of the pulp. (The beans were soaked for thirty minutes in a solution containing 2 ozs. of washing soda to 4 pints of water.) The shape was slightly flat, the shell fitting tight. The interior of the bean was heliotrope to slate blue, and was compact and cheesy. The steamed beans were dark on the outside, showing that the pulp had been difficult to remove. They had a compact and cheesy interior, the colour of which showed various shades of bright violet."

On roasting, the beans treated in this way did not come up to the promising report of Perrot, and they showed the well-known properties of unfermented cacao, being dull purplish in colour, with an inferior odour and an astringent taste.

Granting, therefore, that there may be planters who consider, from their standpoint, that cacao is not improved in quality by fermentation, and who, therefore, not only do not practise this operation, but will even go to greater expense to avoid it, the chocolate manufacturer is in no way uncertain as to the relative values of fermented and unfermented beans. The market price is sufficient indication of his opinion.

Table VIII. shows some figures, obtained by Harrison \* in his work on fermented and unfermented beans, of especial interest in this place. It is worthy of note, however, that, unlike the results of many other observers, the fat-content of the bean is higher than that of those merely dried.

These few remarks upon the advantages of fermentation would not be complete without reference to the work of Fickendey† who, like Perrot, felt convinced that natural fermentation could, with advantage, be replaced by mecha-

\* J. B. Harrison, 1897, from "Cacao," by J. H. Hart, 1900 and 1911.

† Dr. Fickendey in "The Fermentation of Cacao," by H. H. Smith, 1913.

nical and chemical treatment. Fickendey claimed that the most important changes brought about by natural fermentation, with the exception only of development of aroma, can be induced also by the immersion of the cacao in 96 per cent. alcohol or by freezing. Both the browning of the cotyledons and the removal of the bitter taste can be secured by this treatment, after subsequent exposure of the beans to the air, the former being due to the oxidation of the tannin substances to which, in the first place, is attributed the bitterness or astringency. Being confronted with the difficulty of obtaining aroma, Fickendey decided to experiment with fermented beans, and, finding, apparently as a discovery, that the oxidation of the tannin substances increased rapidly in weak alkaline solutions, he proposed that, after the fermentation, the beans should be soaked in a 5—10 per cent. potash solution for ten minutes and subsequently dried. By this treatment he claimed the following advantages:—

- (1) Reduction of the acidity of cacao.
- (2) Increase of activity of the enzymes, enabling them to act more freely and the beans to lose more bitterness.
- (3) Assistance to the cocoa manufacturer in rendering his product more soluble.

TABLE VIII.—*Composition of the Kernels of the Beans of Forastero, Dried, and Fermented and Cured.* (Harrison.)

	Dried.	Fermented and cured.
Water . . . . .	5.000	6.280
1. Albuminoids . . . . .	7.228	6.130
2. Indeterminate nitrogenous matters . . . . .	4.081	2.525
3. Theobromine . . . . .	1.921	1.480
4. Caffeine . . . . .	0.382	0.414
Fat . . . . .	45.831	52.120
Glucose . . . . .	0.247	0.566

# FERMENTATION OF THE BEANS

63

TABLE VIII.—Continued.

	Dried.	Fermented and cured.
Sucrose . . . . .	1·878	<i>nil.</i>
Starch. . . . .	9·048	6·750
Astringent matters . . . . .	7·829	3·470
Pectin, etc. . . . .	2·068	0·770
Cacao-red . . . . .	2·811	2·850
Digestible fibre . . . . .	3·969	5·762
Woody fibre. . . . .	5·435	6·200
Tartaric acid, free. . . . .	0·057	0·420
Acetic acid, free . . . . .	<i>nil.</i>	0·600
Tartaric acid (combined) . . . . .	0·729	0·596
Iron peroxide . . . . .	0·048	0·057
Magnesia . . . . .	0·680	0·621
Lime . . . . .	0·153	0·154
Potash. . . . .	0·951	0·776
Soda . . . . .	0·101	0·196
Silica . . . . .	0·024	0·020
Sulphuric anhydride . . . . .	0·072	Trace
Phosphoric anhydride . . . . .	1·565	1·210
Chlorine . . . . .	0·047	0·048
	<hr/> 99·995	<hr/> 100·000
1. Containing nitrogen . . . . .	1·156	0·980
2.     "     " . . . . .	0·658	0·404
3.     "     " . . . . .	0·406	0·457
4.     "     " . . . . .	0·095	0·119
	<hr/>	<hr/>
Total nitrogen . . . . .	2·810	1·960

That something may be said for Fickendey's contentions can be seen in the sterling quotations given below for five varieties of beans, fermented (1 and 2) and unfermented (3, 4 and 5), of which last, numbers 3 and 4 were treated



by Fickendey's process, and number 5 merely washed and dried. In the cases treated by the Fickendey process, beans from a plantation in Victoria, Cameroons, were submitted to the cooling method without fermentation, by reducing the green beans to a temperature of 0° Centigrade, maintained for three hours. The beans, so produced, were pronounced by a German firm of chocolate manufacturers to possess a good aroma and flavour.

Sample.		London Brokers' Valuation, Feb. 11, 1911.	Liverpool Brokers' Valuation, Feb. 15, 1911.	Manufacturers' Valuation, Feb. 15, 1911.
		Per cwt.	Per cwt.	Per cwt.
No. 1.	Fermented.	About 54s. to 55s.	50s. to 51s.	52s. to 53s.
No. 2.		.. 53s.	49s. to 50s.	52s. to 53s.
No. 3.		.. 52s.	47s. to 48s.	48s.
No. 4.	Fickendey.	.. 53s.	46s. to 47s.	50s.
No. 5.		.. 50s.	45s. to 46s.	46s. to 48s.

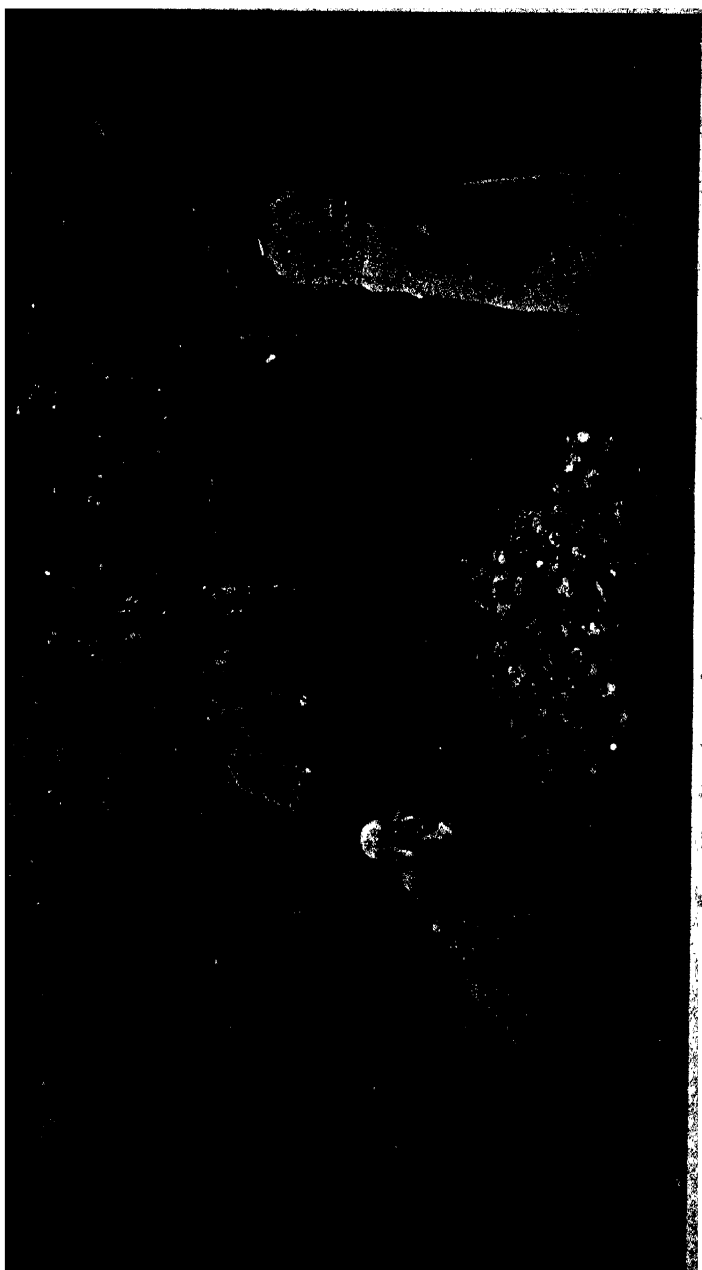
The changes which take place during fermentation will be considered more fully later in this chapter, when the results of analyses of beans, before and after fermentation, are dealt with, and, for the present, it will be assumed that improvement of the kernel and ready removal of adhering pulp are the main objects for which fermentation is employed, the reason why such improvements or advantages are obtained by the process being discussed as necessity arises.

The pods, after they are picked, are sorted into grades according to their quality, the over-ripe, under-ripe and diseased being put on one side.

The grading completed, the pods are "broken" either in the plantation or in the fermenting house, the latter place being preferable owing to the possibility of the decaying husks conveying disease to the trees, if allowed to remain in the plantation.

The shelling or breaking is performed by a cut with a knife on one side of the pod, which is then broken open with the hands (Plate VII), or the more primitive method of





OPENING THE CACAO PODS, TRINIDAD. (See p. 64.)

TABLE IX.—*Changes taking place in the Kernel of the Beans of "Calabacillo," during Fermentation and Curing.*

	Fresh beans.	• Fermented and cured beans.	Loss during fermentation and curing.
Water . . . .	37.637	3.675	33.962
Albuminoids . .	6.696	4.419	2.277
Indeterminate nitroge- nous matters . .	0.531	•	1.521
Theobromine . .	1.352	1.003	0.349
Caffeine . . . .	0.108	0.032	0.076
Fat . . . . .	29.256	29.256	nil
Glucose . . . .	0.991	0.604	0.387
Sucrose . . . .	trace	nil	nil
Starch . . . . .	3.764	3.221	0.543
Astringent matters .	5.004	3.610	1.394
Pectin, etc. . . .	0.657	1.178	+0.521
Cacao red . . . .	2.952	1.390	1.562
Digestible fibre . .	5.112	3.737	1.375
Woody fibre . . .	3.030	2.780	0.250
Tartaric acid (free) .	0.079	0.328	+0.259
Acetic acid (free) .	nil	0.544	+0.544
Tartaric acid (combined)	0.477	0.377	0.100
Iron peroxide . . .	0.032	0.069	+0.037
Magnesia . . . .	0.324	0.375	+0.051
Lime . . . . .	0.054	0.118	+0.064
Potash . . . . .	0.842	0.592	0.250
Soda . . . . .	0.239	0.288	+0.049
Silica . . . . .	0.016	0.022	+0.006
Sulphuric anhydride .	0.079	0.031	0.048
Phosphoric anhydride .	0.749	0.712	0.037
Chlorine . . . .	0.019	0.012	0.007
	<hr/> 100.000 <hr/>	<hr/> 60.442 <hr/>	

TABLE X.—Composition of Sweetings taken from the Runnings of Sweating Boxes, and of Original Pulp.

	Sweetings, Calabacillo and Forastero.	Original pulp, Forastero.
Water . . . . .	84.817	88.030
Albuminoids . . . . .	0.062	1.271
Indeterminate nitrogenous matters . . . . .	0.250	nil
Glucose . . . . .	11.604	0.091
Sucrose . . . . .	0.638	1.001
Astringent matter, etc. . . . .	0.354	0.108
Alcohol . . . . .	0.180	nil
Tartaric acid (free) . . . . .	0.340	0.606
Acetic acid (free) . . . . .	0.892	trace
Acetic acid (combined) . . . . .	0.290	—
Iron peroxide . . . . .	0.038	0.010
Magnesia . . . . .	0.074	0.073
Lime . . . . .	0.029	0.030
Potash . . . . .	0.354	0.248
Soda . . . . .	0.004	0.015
Sulphuric anhydride . . . . .	0.021	0.031
Phosphoric anhydride . . . . .	0.038	0.098
Chlorine . . . . .	0.007	0.061
	100.000	
Tartaric acid (combined) . . . . .		0.351
Theobromine . . . . .		0.348
Caffeine . . . . .		0.059
Fat . . . . .		0.421
Starch . . . . .		1.305
Pectin, etc. . . . .		1.126
Cacao red . . . . .		0.705
Digestible fibre . . . . .		6.564
Woody fibre . . . . .		2.455
Silica . . . . .		0.003
		100.000

dashing the pods off a log of wood, as employed in Nicaragua, may be resorted to.

The beans and pulp are removed from the broken pods and conveyed to the sweating house, where they are allowed "to stew in their own juice" \* in vats (Plate VIII.), until the interiors have assumed the cinnamon-red colour so much desired.

The exact condition to which the beans must be brought by fermentation is known only to the practical planter, but, whether the process is carried out in vats, as in Trinidad and Ceylon, or fermentation is allowed to proceed in the beans packed warm from the sun in sacks, as is the custom of the small grower in Venezuela, the results achieved are the same in their most essential points.

In order to appreciate fully the action taking place during fermentation, it is first necessary to know the composition of the materials employed in the process.

Professor Harrison has made careful analyses of the pulp and beans before and after fermentation, and, though it will be unnecessary to quote in full all his results, the collected figures in Tables IX. and X. will give most of the information required.

The beans and their adhering pulp, during the process of fermentation, rise in temperature from 30° to 40° C. in about three days, finally reaching 48° or 49° C., if a good fermentation has been secured. The fermenting mass should never be allowed to reach a higher temperature than 60° C., and, in order to prevent this, frequent turning of the beans is necessary.

The following results, collected by Preyer, showing the temperature of fermentation in different districts, stated by various observers, are of interest :—

\* Dr. Chittenden in prize essay on "Fermentation of Cacao," *Agricultural Record*, 1890.

	Method of fermentation.	Temperature employed.
Preyer	Heaping method (Ceylon):	
	In the upper half	30—40° C.
	In the lower half	Outside temperature to 30° C.
Chittenden	Some samples fermented well at	26·7° C.
	With the method in vogue in Trinidad	46—49° C.
Morris	By the method practised in the West Indies	About 60° C.
Strickland	Three-tank system (West Indies):	
	In tank 1	Not over 46—49° C.
	In tank 2	Not under 35° C.
	In tank 3	" " "
Friederici	Three-tank system (Cameroon):	
	In tank 1	30—33° C.
	In tank 2	35—38° C.
	In tank 3	Not over 43—45° C.
Preuss	Three-tank system (Surinam)	Never above 45° C.
Wright	Three-tank system (Ceylon):	
	Tank 1	25—30° C.
	Tank 2	30—40° C.
	Tank 3	33—44° C.
Johnson	Heaping method (Gold Coast)	Below 60° C.

In Trinidad, Knapp made the following observations as to temperature in the sweating boxes:—

After	Degrees Cent.	Degrees Fahr.
1 day	30	86
2 days	37	98
3 days	47	117
4 days	48	118
5 days	49	120
6 days	49	120

Hudson,\* at a St. Lucia estate, British West Indies, has carried out a number of experiments on fermentation and has formulated certain axioms, borne out by his experience. Of these, the following are the most interesting:—

(1) The higher the temperature, obtained and maintained for some days by primary natural fermentation, the better

\* G. S. Hudson in "The Fermentation of Cacao," by H. H. Smith, 1913.







SWEATING BOX, TRINIDAD. (See p. 87.)

the class of cacao turned out. (The maximum limit of temperature reached in Hudson's experiments was 119° F.)

(2) The addition of Dr. Lucius Nicholls' pure ferment and yeast food solution induces a higher and more prolonged beneficial fermentation.

(3) The daily "changing" of cacao from box to box by a wooden spade, thereby transferring the bottom seeds to the top, is most necessary to obtain the best results.

(4) Beneficial fermentation ceases as soon as the bottom of the box falls below 100° F.

(5) The larger the quantity of cacao fermented the quicker is a high temperature attained and lost; but small quantities, whilst requiring a longer period, can be fermented just as efficiently as large.

(6) The preservation of heat and moisture by a thick leaf cover is a most necessary part of the process, but, where a pure ferment is not added, the cover can be beneficially omitted from the first and second boxes, to allow the "cacao fly" more free access to the beans, to deposit fermentation germs upon them.

In the same essay, Hudson records temperatures of beans during fermentation, with an average air temperature of 78° F. and the average humidity 79, as ranging between 76° F.—88° F. after three-quarters of a day, and 108° F.—116° F. after 5½ days, when the temperature begins to fall. By using Dr. Nicholls' ferment and solution, under identically the same conditions, the temperature continued to rise up to the end of 6½ days, though his figures do not show higher temperatures than when natural fermentation is allowed to take place, contrary to the opinion expressed in the second axiom.

Preyer,\* in his articles on cacao fermentation, states that the micro-organisms, engaged in the operation, attack the

Axel Preyer, *Woch. für Brau.*, 1901, xviii. (21), 277, and *Der Tropenpflanzer*, 1901, v., and in "The Fermentation of Cacao," by H. H. Smith, 1913.

husk and kill the embryo, whilst the soluble matters and enzymes diffuse through the dead tissue. According to this writer, the fermentation should be stopped when the outer skin is loosened, so as to be readily removable in a subsequent washing process. The same author states that the yeasts, isolated from the vat liquors during a Ceylon fermentation, consisted of budding fungi, similar to *Saccharomyces ellipsoideus* and *S. membranefaciens*, and a new species which he named *S. theobromæ Freyer*, to which he attributes special activity.

The whole process of fermenting and curing may occupy from nine to twelve days, though three days may be sufficient for the first operation, if fermentation starts at once under ideal conditions of temperature.

Needless to say, there can be no set time given for the completion of fermentation, the principle underlying the process being the attainment of certain ends, dependent upon variable conditions. Schulte im Hofe, in an essay in Smith's book on "The Fermentation of Cacao," already frequently mentioned, says, "In the Cameroons it was customary, at first, to allow the cacao to sweat from three to four days, but now the period has been prolonged. In San Thomé the methods employed are more advanced, and the period of fermentation (or sweating) is not always regulated by time, but often by the appearance of the bean, but even here the fermentation is usually allowed to continue too long." The present writer is in agreement with Smith, who has commented on the fact that the San Thomé cacao coming to his notice does not give the impression of being over-fermented.

Schulte im Hofe, Knapp and many others have described the appearance and other characteristics of beans when properly fermented. These may be summed up as follow :—

(1) Change in exterior from almost white to a rich brown,

which should become deep in colour<sup>\*</sup> before removal of the beans from the sweating box.

(2) Change in interior from bluish-violet and mottled to an even reddish-brown tint.

(3) Adhering pulp easy to remove.

(4) Beans become plump and round.

(5) From being dry the interior becomes moist, and, by absorption, the beans become so full of liquid that, when cut, they spurt out juice.

(6) Spaces filled with purple or brown sticky liquid occur between the cotyledons.

(7) The odour of the mass changes from a delicate melon-like aroma to a heavy, sharp fragrance, probably due to ethyl and amyl esters with free acetic acid.

After the process, the skin of the bean is found to be shrunk and toughened and in a fit state to withstand fungoid growths, the kernel being changed from its somewhat purplish hue to a rich brown, and the bitter and astringent taste being greatly modified.

Briefly, the changes which take place in the operation of sweating are as follow :—

The beans and pulp, exposed to the air, are at once attacked by a yeast (*Saccharomyces theobromæ* Preyer), which causes a rise in the temperature of the materials due to the chemical action involved in breaking up the sugary matters subjected to its influence, with the production of alcohol.

Later, acetic acid is developed by the action of bacteria (*Mycoderma aceti*), which speedily attack alcoholic liquids exposed to air. Another increase in temperature is noticed with the second reaction, and so much heat is developed that alcoholic fermentation is practically stopped. Both the alcoholic and acetic acid fermentations are due to the activity of the enzymes or juices secreted by the growing ferments, and it must be inferred, from the analyses given,

that other yeasts and <sup>\*</sup>ferments, possessing proteolytic or albuminoid-splitting enzymes, are also actively engaged in securing the general result of the fermentation process.

The loss of starch from the bean, during fermentation, must be attributed to the diastase content of the *saccharomyces*, as observed by Pfeffer,\* or to the diastase of secretion which all seeds contain, and which plays so important a part in providing soluble nourishment for the growing plant, during the germination of the seed.

The loss of the alkaloid theobromine, during fermentation, has been much disputed. Indeed, Sack has claimed that there is an increase of the theobromine shown, but this statement has been refuted by both Brill and Knapp, and there does not seem to be any scientific support for Sack's contention. Knapp is more emphatic, for he has stated that "I should say definitely that there was no change in the theobromine content, only I am not absolutely satisfied with the delicacy of the methods commonly used for estimating theobromine." With regard to the estimation of theobromine in cacao products, various methods are considered in a later part of the present work.

To recapitulate in proper sequence, the process of fermentation\* is as follows :—

(1) Conversion of starch into soluble dextrin and sugar by means of diastase, an operation which would take place naturally within the bean at the commencement of the fermentation process, even if no *saccharomyces* were present.

(2) Some of the sugars of the pulp, and the original and newly formed sugars of the beans, are next converted into alcohol by the sugar-splitting enzymes of the *saccharomyces*, with a consequent rise of temperature.

(3) The alcohol, so formed, rapidly undergoes fermentation by the agency of *Mycoderma aceti*, with a still further rise of temperature. At the same time, there occurs a loss of

\* Pfeffer, " Ueber die regulatorische Bildung von Diastase "

albuminoid matter, both in the pulp and bean, attributable to the action of proteolytic ferments.

(4) In the presence of air, an oxidation process within the bean, resulting in colour changes due to the action of an oxidase.

The chemical action during the process of fermentation has the effect of withdrawing the soluble matters, contained in the kernel, to the liquid running away from the fermenting mass, and, consequently, the quantities of the soluble mineral matters, chiefly consisting of potash and phosphoric acid, are found to be considerably reduced in the fermented beans and increased in the sweatings.

The astringent matters, to which the acrid taste of the fresh beans are due, are undoubtedly hydrolysed during the fermentation, as they are found by analysis to be in greatly reduced proportions both in the fermented pulp and beans. The relation between astringency and the presence of tannins has already been recorded; that between astringency and correct fermentation is of considerable interest to the chocolate manufacturer.

In the case of the Calabacillo bean, the loss during fermentation and curing in one instance was 62.5 per cent., of which 59.0 was water and 3.5 organic and mineral matters.

Some loss of alkaloids, gums, vegetable fibre, astringent matters and cacao-red occurs in the beans during fermentation, besides the more apparent losses in sugars, starch and albuminoid matters, the only gain of real importance being in acetic acid, the result of fermentation, and certain mineral matters which may be attributed, partially, to the accumulation of dirt.

In later analyses by Professor Harrison of a mixture of beans containing one-fifth Calabacillo and four-fifths Forastero varieties before and after fermentation and curing, the losses and gains of the beans during the process are still more marked, the latter being entirely confined to 0.257

acetic acid, 0.010 iron peroxide, and 0.040 silica, whilst the total loss amounted to 64.062 per cent., of which 56.419 was water, 0.429 sucrose, 1.280 starch, 0.301 glucose, 1.258 astringent matter, 0.982 albuminoids, 1.167 digestible fibre, and 0.270 cacao-red.

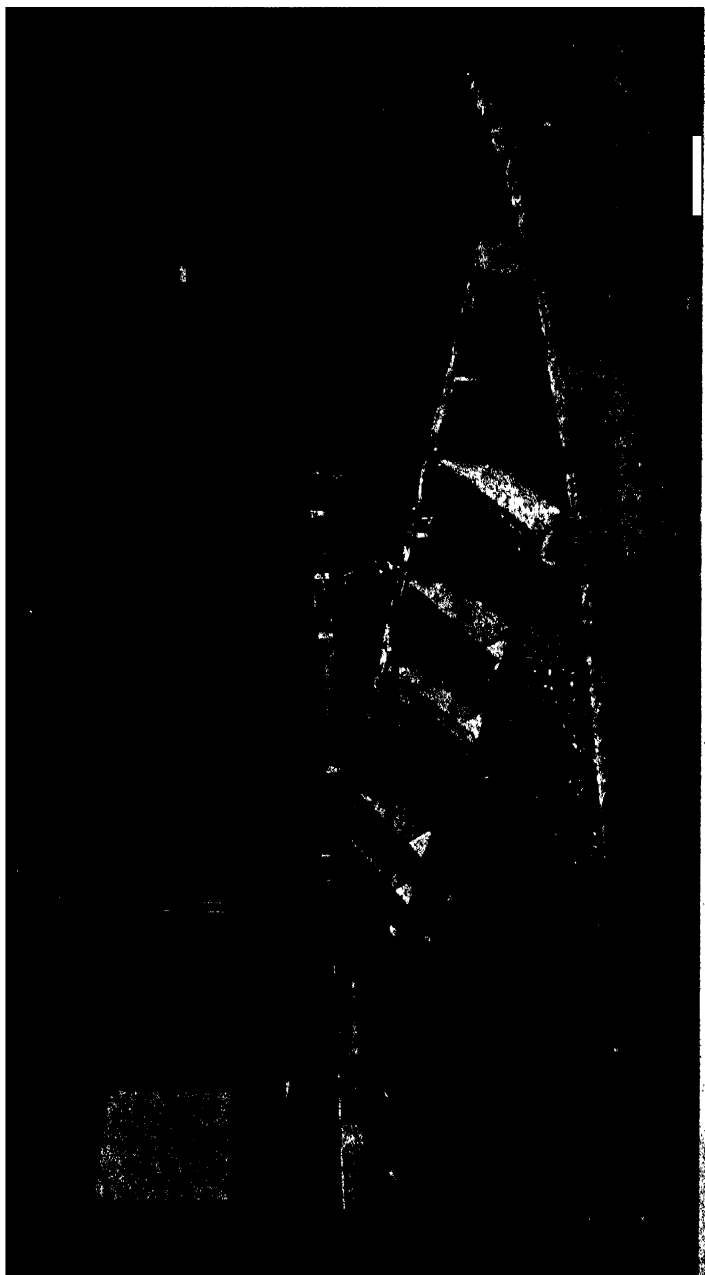
• Before concluding this chapter on fermentation, it is as well to consider one or two aspects that have not as yet been mentioned. There are two distinct processes in cacao fermentation that should be kept strictly separate: (1) the fermentation of the pulp, *i.e.*, the sugary mucilaginous mass in which the beans are embedded, and (2) the changes in the substances of the beans themselves.

The first process of fermentation takes place in the pulp or juice, which contains about 10 per cent. sugar (dextrose and levulose) and 3 per cent. mucin, and which shows an acidity, chiefly due to malic acid, of approximately  $\frac{1}{10}$  N. solution. The specific gravity at 15° C. is about 1.063, and 100 ccs. of juice contain some 0.638 grms. of mineral ash, of which about half the amount is due to equal proportions of calcium and magnesium carbonates. The juice is, then, an excellent medium for the growth of yeasts and bacteria, and it is, of course, due to its fermentation that the rise in temperature is secured, sufficiently high to kill the germ in the bean without injury to the enzymes which play so important a part in the second process.

Many experimenters have suggested that the juice should be used as a source of commercial alcohol or vinegar, and there does not seem to be any practical difficulty in the project. Yet, so far as the present writer is aware, no attempt has been made to place the scheme on a commercial footing, though the subject will be found discussed at some length in many of the technical books and journals dealing with the fermentation of cacao. Smith, in "The Fermentation of Cacao," has gone so far into the matter as to place on paper some calculations which make very good reading,







ARTIFICIALLY DRYING CACAO, TRINIDAD. (See p. 78.)

[Trinidad]

and which accentuate at least the necessity for an intelligent inquiry into the possibility of producing vinegar and alcohol as by-products of the cacao plantation.

Commencing with the world's annual average output of cacao for the years 1910 to 1912 as 227,500 English tons, and the assumption, from Hudson's figures, that 2 gallons of vinegar result from 200 lbs. of cacao, it is clear that 5,096,000 gallons of vinegar could be added to the world's present production. This vinegar, when properly prepared and thoroughly mature and clear, has been described as excellent. "Very occasionally one meets with cacao vinegar that appeals to the palate as the very perfection of vinegar; although it is not deficient in strength its great points are exquisite mellowness and freedom from all bite." But the occurrence of such a fine brew does not appear to be common from the many adverse reports on cacao vinegar that can also be found. The correction of faulty production of vinegar can only be a matter of adjustments of conditions, and, as has been remarked before, there seems no practical hindrance to its successful preparation, save, perhaps, insufficient knowledge of the manufacture of vinegar on the part of the planters, which could be easily remedied.

With regard to the production of alcohol, the literature on the subject seems confused between the manufacture of an alcoholic beverage and that of commercial alcohol. Smith's figures, in the *Preface* to his work, show that the sale of the juice would bring in a total of £127,400 a year, if sold at 3d. per gallon. This total, divided among the planters all over the world, does not seem worth considering, especially as the sale and preparation of the alcohol would entail an alteration to the existing system of curing the beans and the necessity for finding a market for the juice which, presumably, would be used only by local distilleries, when they existed.

It is pointed out by several writers that the cacao juice

fulfils all the conditions necessary to the making of a palatable spirit by distillation and even to making of wine. In Para, we are told, many of the planters pay more attention to producing an intoxicating drink from the green cacao than to the commercial beans themselves. The characteristic flatness of Para beans has been attributed to the squeezing which the beans are made to undergo, in order to free them from the last drop of liquid. No confirmation of this report can be found, however.

The possibility of separating or utilising the pectins from the cacao juice has not been overlooked, as Knapp \* has suggested. A cacao jelly is occasionally made from the juice by adding an equal bulk of sugar. Its manufacture is very similar to that of guava jelly, but the luscious flavour of the latter cannot be approached by the rather insipid flatness characteristic of "Cacao jelly."

\* A. W. Knapp, *Tropical Life*, x., pp. 42, et seq.

## CHAPTER VII

### PREPARATION OF THE BEANS FOR MARKET: WASHING, DRYING, FINISHING OR POLISHING

#### **Washing.**

AFTER fermentation is complete and the required colour and aroma of the beans obtained, the cacao should be submitted to washing at once, if at all; otherwise fermentation will proceed beyond the desired limits.

No harm whatever can come to the cacao by washing, from the points of view of the chocolate manufacturer and consumer, though the loss of weight attending the process, due to the removal of adhering pulp, may be imagined by the planter to be a matter of considerable importance and will frequently cause him to forego the operation. Cleaned beans will always command the higher price, and, in these days of scientific enlightenment, there is little to be gained by the planter in neglecting to wash the cacao, since the absolute yield or weight of the kernel, apart from the skin and its adherent dirt, is as important a factor in determining market value as the quality of the kernel itself.

The loss from washing has been variously estimated. Sack \* has stated that, in Surinam, a loss of as much as 15 to 18 per cent. is admitted, but his own figures show 4 to 5 per cent. from well fermented beans and up to 8 per cent. for dirty cacao, figures which compare more closely with Preuss' † figure of 4 per cent. and Eigen's ‡ value of

\* J Sack in "The Fermentation of Cacao," by H. H. Smith, 1913.

† Preuss, "Reise nach Central und Sud-Amerika," 1901.

‡ Eigen, *Der Tropenpflanzer*, February, 1903.

6 per cent., both for Cameroon cacao. Schulte im Hofe has told us that, in the Cameroons, "at first . . . the cacao was washed after fermentation had been completed, whereby the remains of the fruit pulp adhering to the beans were removed. As in chocolate factories the shell is removed from the kernel in any case, it is immaterial whether some of the fruit pulp still adheres or not. This fact was soon recognised, and now the beans are not washed but generally dried immediately after fermentation." There is no need to comment upon this clear indifference to the finish of the product and to the real needs of the chocolate manufacturer, especially at the present time with high freight rates. The advantages of washed beans to the cocoa and chocolate manufacturers can be seen when the returns of "husk" and "nib" from different beans and the price obtainable for the winnowed "husk," the manufacturer's by-product, are discussed.

The beans should be placed in sieves or troughs and thoroughly stirred and scrubbed under water, till all adhering organic matter is removed and the beans are clean and smooth, when they are ready for drying.

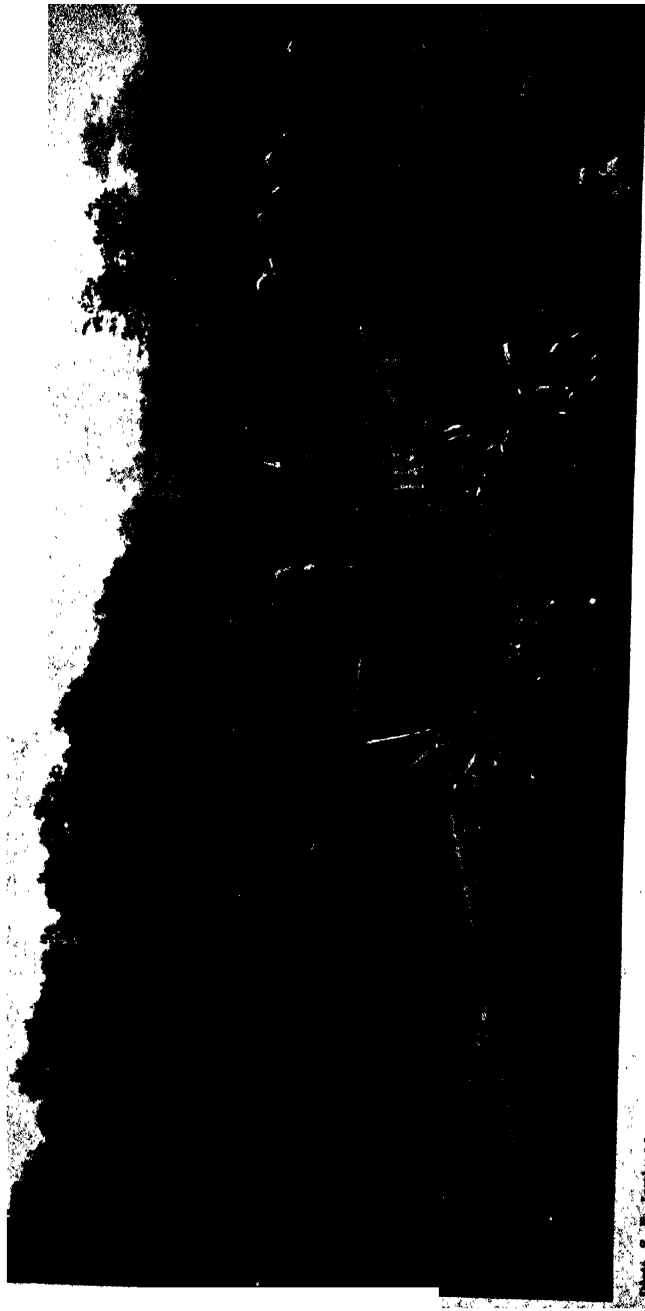
#### Drying.

The method adopted for drying may be by exposure of the washed cacao to the heat of the sun or by a system of hot water or steam pipes under cover, if the climate does not lend itself to the open-air process (Plates IX. and X.).

In Trinidad, the beans, which are not always washed, are exposed to the heat of the sun on open wooden floors or trays, which are covered up, when it rains, either by a movable roof or by placing the trays under a fixed shelter.

In Ceylon, the washed beans are dried either artificially or by the sun's heat, as slowly as will allow the danger of the formation of mould to be avoided, a process accelerated, without injury to the quality of the cacao, if proper fermentation has been accomplished.





Photo, S. E. Tappan.

CACAO BEANS DRYING IN THE SUN, TRINIDAD. (See p. 78.)

[Trinidad]

The beans, which are usually spread on the drying floors to a depth of 3 or 4 inches, must be kept constantly turned, to prevent local over-heating and to enable a circulation of air to reach all parts of the layers.

The best cacao is undoubtedly obtained by sun-drying. It is hardly possible, however, to do without drying plants, except in a few localities, and there is no practical objection to their use. Opinion differs as to the best plants for the purpose. Those constructed on the principle of bringing the beans into contact with opposing currents of air seem to be the cheapest to work, but are open to the objection that the cacao dries irregularly. Unless the mass is stirred whilst drying, the beans adhere together, and, at the points of contact the drying ceases or becomes delayed, and moulds are encouraged. The most practical method is that shown in the illustration (Plate IX.).

Hudson, in the essay already mentioned, says: "I am strongly of opinion that the ease, economy and certainty of drying cacao *in vacuo* will eventually cause it to supersede all the methods on estates producing 100 bags or more annually." Experiments, made then (1908), certainly justify his contention, though the present writer has not heard of any other developments towards carrying this method into practice. The "Passburg" system of drying *in vacuo*, which we are aware has been eminently successful in drying rubber, fruits and vegetables in recent years, is advertised for drying cacao beans also, and there seems no obvious objection to its use for this purpose, except that the volatile ethers, which it is required to retain, might be more easily drawn from the interior of the bean than by the older method of slow-drying.

Bainbridge and Davies\* have thrown a little light on the probable advantages of slow-drying by emphasising the

\* Bainbridge and Davies, "The Essential Oil of Cacao," *Jour. Soc. Chem. Ind.*, November, 1942



fact that a number of possible ethereal substances are added to the essential oil of cacao during fermentation. These ethers, formed in the fermenting juice, penetrate the husk and enter with other liquids, during the fluctuating changes of temperature, into the interior of the beans. At least, the less volatile of these ethers will be retained within the bean, if subsequent drying is sufficiently slow. This hypothesis was confirmed when, later, the analyses of the essential oil of cacao were made, showing that the earlier fractions, distilled, were rich in esters of the order expected, and analogous to those occurring in other spontaneous fruit-fermentation taking place at comparatively high temperatures in the presence of a free supply of air.

The whole question of drying of cacao has been well reviewed by Hudson, whose original work should be consulted, if further information be required.

The process of drying can be summed up in the words of Schulte im Hofe: "Whatever system may be selected, the main thing always is to use the lowest possible temperature for drying, but, above all, any further acidification, or the formation of butyric acid and the development of moulds, must be avoided."

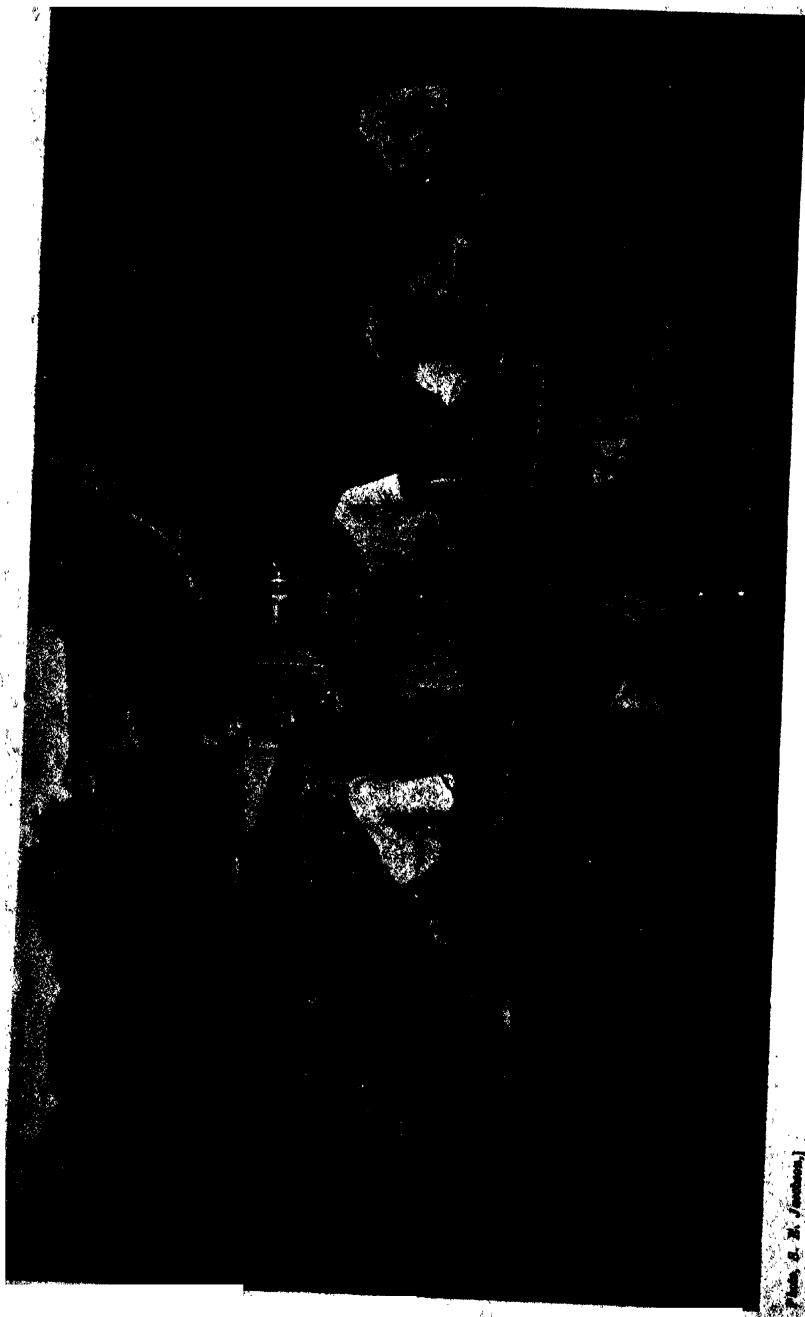
### Finishing.

In order to accelerate the drying of the beans without applying extra heat, especially during the damp weather, the planters of Trinidad, Venezuela and certain other countries make use of fine dried earth, which they sprinkle over the beans and intimately mix in with the drying mass (Plate XI.).

This provides a ready method of removing any remaining pulp, as the drying beans are frequently raked over, and the friction, of one bean rubbing against the other, soon causes the adhering matter to fall away. Other purposes which are served by this treatment are an improved finish in the



PLATE XI.



“CLAYING” CACAO, TRINIDAD. (See p. 80.)

**APPENDIX I**

Photo. B. W. Jackson, I

appearance of the beans, protection against mould, and additional weight.

With regard to these secondary advantages, the beans, treated with the red earth or clay that is usually employed, certainly present a brighter and cleaner appearance, and the mineral covering, so formed, provides additional protection against mould and fungus growths, so feared by the cacao planter, which frequently necessitate the beans being dried for two or three days beyond the time usually allowed by the planter for complete drying. There can, however, be no real advantage to the planter in adding weight to the beans, since the market is wide awake to the possibilities of sophistication.

It is, perhaps, significant that of late years, in this country at any rate, less "clayed" cacao has found its way into the market. Venezuelan cacao shows a decided tendency towards better washing and less clay, and one is inclined to look to-day with suspicion upon "clayed" cacaos which formerly might have been said to carry the hall-mark of quality. Both planter and manufacturer now possess scientific advisers. Hudson, in his essay, bemoaned the fact that "while one cannot doubt that the large cacao buyers in Europe and the United States are trained, keen experts at their trade, it is a curious point to the planter that they will pay 2s. per cwt. more for 'polished' or 'clayed' cacao, which only improves the superficial appearance and adds to the weight without improving the quality in any appreciable degree, and yet can only be induced to give the same advance on ordinary prices for 'washed' cacao, which, according to general observation, frees the beans from gums and other extraneous matter (which should be of no use to the manufacturer) to the extent of from 4 per cent. to 5 per cent. in dry weight." We do not like the implication contained in this paragraph, nor the assumption that "the testa or seed-skin with its adherent

matter is used indifferently with the seed contents in manufacturing chocolate or cocoa," yet we are aware that there has been, and still unfortunately is, some grain of truth in his suggestion. But we cannot all be "tarred with the same brush," and the present writer knows of more than one manufacturer, both in England and in the United States, who positively refuses to pay for "clayed" cacao on the ground of extra weight which is of no value. Besides, if clay is to be added to cocoa and chocolate, why not add it in quantities that make it worth the while? Of course, Hudson's suggestion is really nonsensical, for the bulk of chocolate manufacturers certainly do not adopt this method of adulteration, which, besides being primitive, is open to the objections of easy detection and the production of gritty goods. "Clayed" cacaos of Ceylon, Central America and Samoa are, moreover, more often than not, beans of the highest quality, being of the Criollo or thin-skinned variety which would, in any case, command higher prices than beans of Forastero or Calabacillo origin for the excellence of their aroma and their high fat-content.

Excessive "claying" has, however, been practised, and the Board of Agriculture of Trinidad caused posters to be placed in the colony, early in 1914, to the effect that "if excessive 'claying' be not discontinued, it will be necessary in the general interest of the colony to bring this matter to the notice of the Government, with a view to the introduction of legislation making excessive 'claying' a punishable offence." Originally, the use of clay was considered to be the best means of preserving cacao beans, and, without doubt, its use materially renders the husk impervious to moisture, and so prevents decay and the growth of moulds, whilst assisting in retaining the cacao aroma. In addition, the uniformity of colour, produced by rubbing the beans in clay, adds considerably to the appearance of the sample. It has been already pointed out, however, that "claying"

is, to-day, no criterion of quality, and the beans coming into the market are judged for their flavour and aroma, the percentage of fat the cotyledons contain, and the "return" from a given weight of beans—that is to say, the amount of "nibs" or cotyledons, obtained from a given weight of the raw beans, which should, alone of the cacao beans, be used in the manufacture of chocolate and other cocoa preparations.

The quantity of clay permissible is, of course, impossible to give. Hart suggests about 3 lbs. to the barrel of cacao beans or about 1 lb. to every 100 lbs. of wet cacao, but, clearly, the quantity, used, must depend upon the needs of the planter in the first place, and, secondly, upon the degree of dryness to which the wet beans have already attained before "claying" is commenced.

Olivieri \* has given figures of  $\frac{1}{4}$  to  $\frac{1}{2}$  lb. of red ferruginous earth per barrel (110 lbs.) of wet cacao.

Buyers are, to-day, certainly inclined to give less for "clayed" cacao than formerly, as they realise that the earth is dead loss to them. Bannister † found that the integument of "clayed" cacao beans from Trinidad contained as much as 5.12 per cent. of sand and 2.87 per cent. of silica. As a general rule, the amount of clay on "clayed" cacaos does not exceed 2 per cent. of the commercial beans.

Quoting from Johnson's book on cocoa (1912), the present writer is in entire agreement with two of the largest cocoa and chocolate manufacturers in this country who, in reply to some questions put to them regarding treated cacao beans, said: "We consider the preparation for the market of by far the largest proportion of Bahia, Trinidad, Grenada, San Thomé and Kameroun cocoa is perfectly satisfactory to the consumer, and

\* Olivieri, "Treatise on Cacao"

† Bannister, *Jour. Roy Soc. Arts*, 1890.

dislike any tampering with the bean as by washing, claying, oiling, etc.," and again, "In buying cocoa we certainly do go into the question of loss of weight by moisture and shell, and have carefully worked out a table of the various cacaos showing their different losses. In many cases the loss through moisture and shell amounts to some 25 lbs. and over per cwt."

### Polishing.

\* In certain countries, even where the use of earth is customary, the fear that mildew should attack the drying bean is so great that a process of "hand-rubbing," or "dancing," is sometimes employed (Plate XII.).

"Dancing" consists of treading the layers of drying beans with the naked feet and, as with "hand-rubbing," serves the double purpose of removing adhering matter and mildew from the beans, by friction of one against another, and of giving a finish or polish.

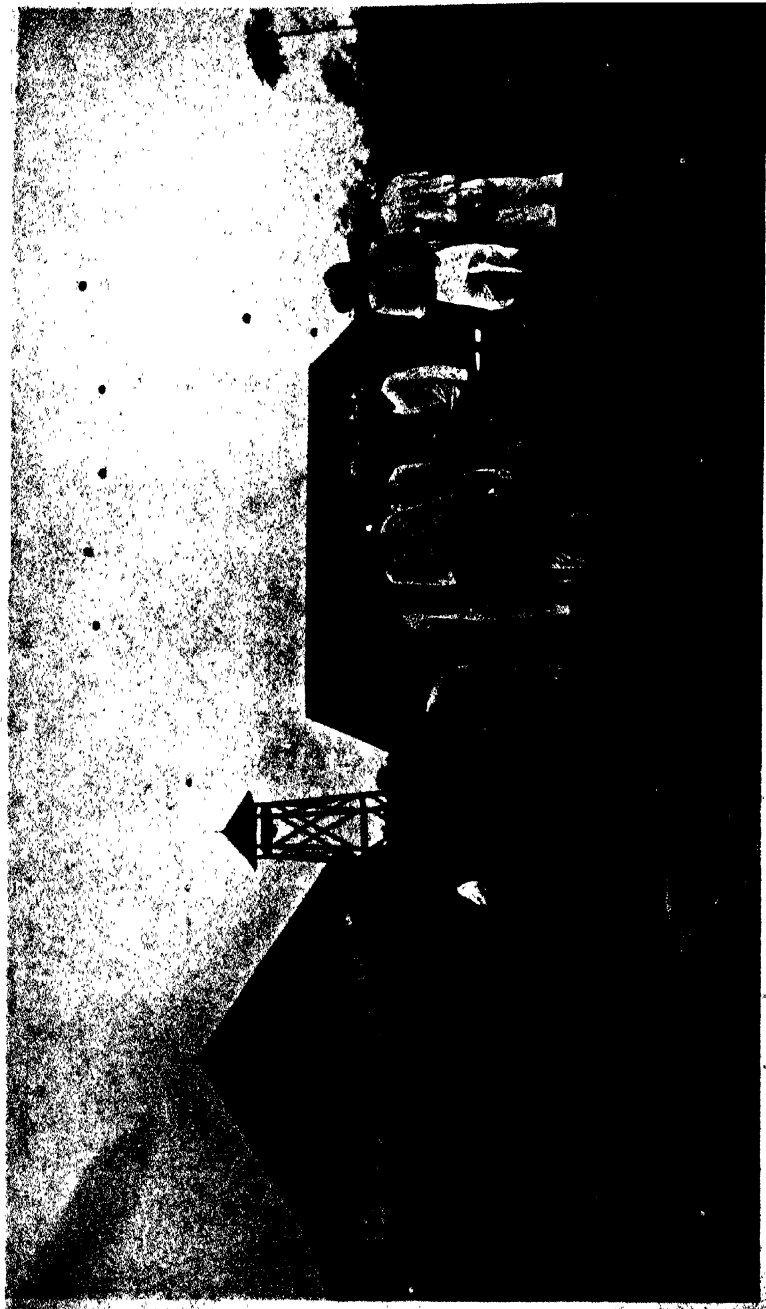
It is difficult for anyone but an experienced person to say when the cacao has received sufficient drying, but, from analyses of the unroasted shells and kernels\* of beans bought on the market, it would appear that the cacao may be considered sufficiently dry when the moisture content of the shells has been reduced so as not to exceed 18 per cent., and of the kernels 8·5 per cent. These figures, given by Zipperer, are undoubtedly excessive, and the present writer has seldom handled beans with more than 8 per cent. of moisture, which should certainly not be exceeded. The amount of moisture in the kernels or "nibs" has been variously given: 5·23 per cent. (Trinidad), 4·75 per cent. to 4·90 per cent. (Forastero-Amelonado), 4·20 per cent. to 5·40 per cent. (Caracas), 4·50 per cent. to 6·50 per cent. (Forastero-Cundeamor "nibs"), 4·55 per cent. to 5·00 per

\* Zipperer, "Untersuchungen über Kakao, etc.," 55, 56, 57





PLATE XII.



[Rev. A. H. Jacobsen]

"DANCING" THE CACAO, TRINIDAD. (See p. 84.)

[Trinidad.]

cent. (Gold Coast), etc. Figures as high as 12 per cent. have been recorded by Playfair and Lankester and 11.60 per cent. by Boussingault, but such large figures as these are very unusual.

A good deal of care is necessary during the operation of "polishing." The beans, from continual friction, assume "a polished Spanish mahogany" appearance, if correctly "danced," according to Hart. When the beans appear in this state, the heaps should be opened out, and drying should be allowed to proceed as rapidly as possible. On the following day, the heaps are again "danced," which, with the drying heat of the sun, fixes the polish upon the skin of the bean.

Rapidity of drying after this stage determines the degree with which the polish is fixed, and Hart recommends treatment in a rotary dryer.

Patent automatic polishers, one by Barnard and another called the "Malins-Smith" cacao polishing machine, are described by Hart, and there can be no possible difficulty in securing the desired result by machine, since the operation is essentially to seal up all possible cracks and crevices in the husk of the beans, to strengthen the shell and to preserve it from cracking during transit to the market, processes which can be conducted as efficiently by machine as by hand.

Any accusation of a desire to give weight to the beans by "claying" must be removed if "polishing" is also adopted, for, though, of course, some clay is worked into the husk, the majority of it is rubbed off by "polishing." As Hart has pointed out, some countries prefer to wash the beans instead of "claying" and "polishing." It matters little which process is adopted, provided that each is scientifically controlled, and that the beans come into the market as the cocoa and chocolate manufacturer requires.

## RÉSUMÉ OF CHAPTERS IV. TO VII. INCLUSIVE.

IMPORTANCE OF NATURAL CONDITIONS AND PROCESSES OF  
FERMENTING AND CURING ON QUANTITY AND QUALITY  
OF THE FINISHED CACAO.**Natural Conditions.**

Site, soil and seed play very important parts in determining both the quality and quantity of the resulting beans, inasmuch as they constitute those natural conditions which regulate the production of good healthy crops of fixed varieties of cacao.

The site must be one sheltered from prevailing winds and shaded from the direct rays of the sun, both conditions being accomplished by selecting a plantation in the shadow and under the lee of high hills or mountains, or by erecting artificial shelters and growing shade trees to serve the double purpose.

The soil should be of good light loam, of reasonable depth, and should contain sufficient quantities of nitrogen, lime, potassium salts and phosphoric anhydride.

Good drainage and freedom of the soil from salt are most essential to the successful culture of cacao.

In the absence of any or all of the necessary components of the soil, manures to supply the deficiency should be applied. Stable manure, when obtainable, is most to be recommended for its safe application and efficiency.

If these conditions of site and soil are observed in districts, where the temperature never falls below 69° F., and which possess a normal rainfall, a good acreage yield is assured.

The seed is one great factor in determining quality and may be chosen from Criollo, Forastero, or Calabacillo varieties of cacao.

Criollo cacaos give beans of the finest quality, but the trees are not hardy and can only be successfully grown under the best conditions of climate and in the richest of soils.

Calabacillo cacaos are the strongest growers and can be made to succeed in very poor soils, but the beans are of inferior quality and never command the highest market price owing to their astringent and bitter taste.

Forastero cacaos occupy an intermediate position between Criollo and Calabacillo and may be expected to flourish wherever cacao can be reasonably expected to grow.

Hart recommends the grafting of Criollo on to the Calabacillo variety, thus obtaining the finer qualities of both kinds and ensuring a regular crop of beans of constant species.

Pruning is necessary for production of luxuriant foliage and good yield of fruit, but, where incisions are made, anti-septic dressings should be applied to the wounds, in order to prevent the access of parasitic fungi and insects.

Disease may be avoided by keeping the trees healthy and strong, and by paying attention to the soil of the plantation and the proximity of diseased shade trees, and may be held in check by spraying the affected parts with solutions of sulphate of copper or iron. After shelling, the pods should not be allowed to rot within the precincts of the plantation, and all diseased branches, pods and other parts of the tree should be burnt.

In picking, care should be taken to prevent injury to the flowers of the next crop, which occur at the point of juncture of the pod with the tree, and that only ripe pods are picked, if the finest quality cacao is required.

Shelling should be conducted so that no injury occurs to the enclosed beans, and the precaution observed that no diseased pods be allowed to rot near the plantation.

### Processes of Fermenting and Curing.

Fermentation commences with the conversion of some starch by diastase into soluble starch and sugar, followed by alcoholic and acetic fermentations which reduce the proportion of sugars in the bean.

The process of fermentation operates both on the pulp and the beans. In the pulp, alcoholic fermentation raises the temperature and is followed by acetic fermentation. The rise of temperature is sufficient to kill the germ without preventing the action of the enzymes which are contained within the bean and necessary to bring about the changes in aroma and colour.

The benefit to the beans, brought about by fermentation, may be simulated by other processes, such as heat, treatment with alkali solutions, etc., but, so far, fermentation has been found to be the simplest and most efficient method of bringing about the desired result on the plantation, even though it may be less easy of scientific control than the other processes suggested.

During fermentation, the beans lose a certain amount of moisture, albuminoid matters, alkaloids, gums, fibrous matters and cacao-red, besides the greater losses of starch and sugars.

The best temperature for fermentation is about 49° C., and 60° C. should never be exceeded.\* The whole process may occupy from nine to twelve days, though three days may be sufficient under ideal conditions.

The most obvious advantages gained from the process of fermentation and curing are: (1) To improve the quality of the kernel by loss of astringent matters; (2) the modification of the colour of the kernel from purple to a fine cinna-

\* Preyer, in *Der Tropenpflanzer*, 1901, points out that, in Ceylon, the most favourable temperature for fermentation is 38° C., above which inferior results are obtained. This, however, is not universally accepted as the "optimum" temperature. *Vide also Chapter VI.*

mon-brown; (3) to render the removal of adhering pulp more easy, enabling the beans to be more quickly dried; and (4) to toughen the skin of the bean so as to make it more impervious to mildew.

It is advisable to wash the beans immediately after fermentation to prevent further action, and to regulate the drying, so that the maximum time is taken without risk of the formation of mildew.

It seems to be unanimously agreed that slow drying is desirable, though only slight chemical explanation is forthcoming.

To prevent mildew from attacking the drying cacao, it is customary in many countries to sprinkle fine dry earth, usually of a red and ferruginous nature, upon the beans. This operation also gives a better appearance to the beans and removes the remains of any adhering pulp.

In some countries, "hand-rubbing" or "dancing" is employed in order to remove adhering matter and mildew from the drying beans, by friction one with another, and to give a fine polished appearance to the cacao.

## CHAPTER VIII

### CHARACTERISTICS OF THE PRINCIPAL KINDS OF COMMERCIAL CACAO BEANS

IN the foregoing chapters, it has been shown how the appearance of the beans, prepared for market, is affected by the variety of cacao grown and by the varying systems of fermentation, drying, finishing and polishing, employed in the different cacao-producing districts.

By means of these external differences in the finished beans, it is possible for the purchaser to tell, with practice, the land of their origin and, taking into consideration the appearance and flavour of the internal kernel, to determine the nature of the variety of cacao grown and of the processes which it has undergone in preparation for the market.

In considering the descriptions of the beans which follow, it must be borne in mind that blends may be found on the market and that seasonal differences, especially during the harvesting of the bean, may be responsible for variations in appearance and flavour from the general characteristics here given.

The cacaos on the market are of either American, West Indian, African or Asiatic origin. American cacaos include those from Ecuador (Esmeraldas, Guayaquil Arriba, and Guayaquil Machala); Venezuela (Caracas, Maracaibo, Carupano, La Guayra, and Puerto Cabello); Surinam (Berbice and Essequibo); Brazil (Para, Maranhão and Bahia), and Cuba.

The cacaos of most importance derived from the West Indies include those of Haiti, San Domingo (Samaná), Trinidad, Grenada and Jamaica.





PLATE XIII.



SORTING CACAO BEANS ON A DRYING FLOOR, TRINIDAD.

[Trinidad.]

[Photo, S. B. Jackson.]

From Africa come the cacaos of San Thomé, Cameroon, Gold Coast (Accra) and Fernando Po.

The Asiatic cacaos of most importance are from Ceylon and the Dutch East Indies (Java).

The *Gordian* divides cacaos into two groups, thus :—

*Fine sorts or "Edelkakao."*—Venezuela, Ecuador, Trinidad, Grenada, Ceylon, Java, Jamaica, Surinam and Lesser Antilles.

*Ordinary sorts, or "Mittelkakao."*—Bahia, Kamerun, San Thomé, Samaná, Accra and Haiti.

In 1914, Surinam appeared as "Mittelkakao" with Gold Coast, Para, Lagos, etc.; whilst Greater Antilles appeared as "Edelkakao."

The production of the fine sorts throughout the cacao-producing countries has fallen off from a figure of 79 per cent. of the total in 1895 to 32 per cent. of the total in 1912. In the latter year, 32 per cent. represented 72,042,000 kilograms and the total 226,619,000 kilograms, the total output of the two kinds in 1895 being given as 76,213,000 kilograms. In 1909, of the total cacao consumed in America, 54 per cent. were "ordinary" sorts, in Germany 75 per cent., in France 48 per cent., and in England 46 per cent. The average prices during 1909, 1910 and 1911 on the Hamburg market are shown as follow :—

*Prices in Marks per 50 Kilograms net (1 mark = 1s.)*

	1909.		1910		1911.
Summer Arriba.	66	.	65	.	61½
Trinidad .	56—58	.	55—57	.	57—59
Kamerun .	51	.	50	.	54
Bahia .	51—55	.	50—53	.	51—56
San Thomé .	47—52	.	46—50	.	51—54½
Samaná .	49	.	47½	.	51
Accra .	47	.	47	.	48—50
Haiti .	45	.	44	.	45

**American Cacaos. •**

ECUADOR { 1914 output, 41,451 tons.  
 { 1917 output, 40,000 tons (estimated).

(1) *Esmeraldas*.—This district, in 1914, produced about 75 tons of cacao which is known by the name “Esmeraldas.” This is the only cacao, not of Forastero origin, grown in Ecuador. This variety is of finer and nobler qualities than other cacaos grown in the province and more closely resembles Venezuela Criollo. Esmeraldas cacaos are seldom found on the market and are mostly consumed locally.

Beans : Small, heavy, plump, almost round but slightly olive-shaped. Shell : Yellow-brown of unequal colour. Kernel : Dark violet. Flavour : Mild and fine.

(2) *Guayaquil Arriba or Arriba*.—Grown in the province of Los Rios, which produced, in 1914, about 25,000 tons. The main crop is reaped between March and June and is of Forastero origin, variety Amelonado. It is generally recognised that the best qualities of Arriba are only slightly fermented, which necessitates a very thorough drying, if the best product is to be secured. The drying is conducted entirely by the aid of the sun, and earth or clay is not used, with the results that drying is often very incomplete and that mouldy Ecuador cacao is not uncommonly found on the market. “Prime red Summer Arriba” cacao is of the finest quality and is principally sent to European markets, Germany and Holland taking the greater portion before the war. America has, however, increased her consumption of Arriba cacao considerably in recent years.

Beans : Flat, irregular, of unequal colour. Shell : A light brown, lighter than most Ecuador cacao. Kernel : Dark on outside, lighter towards centre. Flavour : Fine, somewhat scented and sweet to the palate.

(3) *Guayaquil Machala*.—A very variable cacao, of less value and of lower quality than “Arriba,” grown in the

province of El Oro, producing about 3,500 tons annually. The main crop is gathered in September and October, but is also taken May to October. Like the "Arriba," it is of Forastero origin and Amelonado variety, the differences in flavour, appearance and consistency between Arriba, Machala and Balao cacaos, the last named grown in the province of Guayas, producing about 6,000 tons annually, being due, respectively, to the differences in the soil and climate of their provinces of origin. The beans are slightly more bitter than Arriba cacaos. This cacao has large sales in America at the present time.

Beans: Flat and irregular, smaller than "Arriba."  
 Shell: Similar to "Arriba," but slightly darker. Kernel: Similar characteristics to "Arriba," but less uniform. Flavour: Pronounced chocolate flavour, less delicate and more bitter than "Arriba."

Besides Balao cacao, already mentioned, there is another produced in Ecuador, named "Manabi" from the province in which it is grown. Some 2,000 tons are grown annually. "Manabi" usually commands a lower price than Balao or Machala cacao.

VENEZUELA	{ 1914 output, 17,606 tons.
	{ 1917 output, 19,722 tons.

(1) *Caracas*.—Grown in the eastern part of Venezuela, this cacao is of very fine quality, though Puerto Cabello and Maracaibo cacaos often fetch higher and more fancy prices. The beans come upon the market classified as Chuao, Ocumare, Choroní, Guiria, Río Caribí, Río Chico, etc., and often as Puerto Cabello, Carupano and La Guayra, though the latter three should be kept distinct as far as possible. The confusion of names arises both from the districts in which the cacao is grown and from the ports of shipment. Caracas cacao may be divided into two classes: (1) small-beaned, and (2) large-beaned—the latter being of Criollo

origin, the former Forastero and similar to Carupano cacao. The beans are nearly always "clayed," to avoid mould, and, consequently, appear clean and of brick-red hue. A great part of the Caracas cacao goes to France and Spain, though both England and the United States have learned to appreciate the fine quality of its flavour.

Beans: Irregular, but large and olive-shaped. Shell: Of the smaller varieties, thick, of the larger varieties, thin, covered with smooth reddish brown earth. Kernel: Pale brown to rich brown colour. Flavour: Fine and full, with pronounced chocolate aroma.

(2) *Maracaibo*.—The cacao shipped from the port of Maracaibo is, correctly speaking, Caracas cacao or Surinam cacao, but it should be of the highest quality grown in the western portion of Venezuela, whence come the best cacaos. The production of "Maracaibo" is not large, and the best of it goes to the French and Swiss chocolate manufacturers. It is mostly of Criollo origin.

Beans: Medium to large, irregular, similar to "Caracas." Shell: Smooth, covered with reddish brown earth. Kernel: Red-brown in colour. Flavour: Strong, somewhat harsh to the palate, but of pronounced chocolate aroma.

(3) *Carupano*.—Mostly grown in the eastern provinces round Cariaco, Carupano, Rio Caribe, Aragua, etc., and along the Orinoco, of which Ciudad Bolivar forms the centre. This cacao is of inferior quality, of Forastero origin, and it is mainly shipped from the port of Carupano. In 1909, the export of Carupano cacao was over 4½ million kilograms.

Beans: Flat and smaller than "Caracas." Shell: "Unclayed" and similar in appearance to "Trinidad," of a dark colour. Kernel: Red-brown to purple in colour, according to degree of fermentation. Flavour: Often sour to smell and bitter to the palate, yet, after roasting, of good chocolate flavour.

(4) *La Guayra*.—Some of the best cacao on the market is shipped from the port La Guayra. This variety should be almost entirely of Criollo origin, cultivated in the valleys near the coast. In 1909, the export of La Guayra cacaos was more than 8½ million kilograms.

Beans: Full and plump. Shell: Similar to large Caracas cacao, reddish brown or dark orange in colour. Kernel: Rich brown. Flavour: Sweet, nutty and of pronounced chocolate aroma.

(5) *Puerto Cabello*.—Grown in the west of Venezuela, this cacao, like “La Guayra,” should be of the finest quality and should command the highest prices. A certain quantity of large Caracas cacao, grown in Chuao, Choroní, Ocumare and other places between Puerto Cabello and La Guayra, is shipped from Puerto Cabello and passes under that name. Between Puerto Cabello and La Guayra is grown the pure Criollo cacao, and the product from these parts is the finest in the world. The exports from Puerto Cabello, in 1909, were between two and three million kilograms. The description of the beans is similar to that of “La Guayra.”

SURINAM 1911 output, 2,806 tons.  
1913 output, 2,978 tons.

The quantities of Surinam cacao on the market are comparatively small, though increasing. The *Gordian* classified it as “Mittelkakao.” The cacao, after fermenting, is immediately dried without washing, a fact which accounts for the poor appearance of Surinam beans, seen by the present writer. The point has been raised by Dr. J. Sack and others that buyers were prepared to pay only 2 cents. per kilo more for washed than for unwashed cacao in the market, and that, with the low price of 55 to 60 cents (1913), the process of washing did not pay the planter.

The beans are mostly “clayed,” but often with what appears to be dark earth or cinders, and the beans, examined,

have been of rather inferior quality, which may be classed with Carupano or low Trinidad cacaos.

Dutch Guiana has doubled its output between 1912 and 1917, the former year showing 9,500 tons, the latter 19,000 tons, of which the majority went to Holland, England importing, in 1912, some 50 tons and, in 1915, 500 tons.

BRITISH GUIANA { 1914 output, 23 tons.  
                          { 1917 output, 3.5 tons.

The variety of cacao cultivated is Forastero, and, from recent examples of beans examined, there does not seem to have been any improvement in the methods of preparation. This is doubtless due to the facts that cacao-growing is an industry for small farmers only and that, owing to the difficulties of transport and labour, improvements in the system existing and a more studied effort to secure uniform products would require considerable capital. Otherwise, the product might be made at least of second grade quality.

The two principal districts are Berbice and Essequibo, and, though the writer is given to understand that both banks of the Berbice River provide equally good land for cacao, the Essequibo cacao, examined, seems to be of better quality.

The general description of British Guiana cacao is :—

Beans : Small to large, very variable ; “ Essequibo,” the larger sample examined. Shell : Covered with grayish earth, easily removed from the nibs. Kernel : Dark brown or purple, very variable. Flavour : Harsh and smoky to the palate.

BRAZIL { 1914 output, 40,112 tons.  
                  { 1917 output, 54,728 tons.

(1) *Para or Para Maranhão*.—The output of Para cacao has remained nearly stationary, varying between three million and five million kilograms since 1894. Bahia cacao, on the other hand, has showed a fairly steady increase. The

natural conditions of Brazil are even more favourable for cacao-growing than in Ecuador, but the methods of cultivation and preparation are more primitive, and, consequently, the products are both poorer and more unequal in quality. Para cacaos are more high-priced than "Bahia," and large quantities go to France.

Beans : Fairly plump, of dark reddish colour, but small. Shell : "Unclayed" and red-brown. Kernel : From chocolate-brown to purple, according to the degree of fermentation. Flavour : Sweet and aromatic, when correctly fermented and gathered ripe, but often harsh and bitter, when these precautions have not been observed.

(2) *Bahia*. - This is often confused in name with the Bahia cacao of Ecuador, called also "Manabi," "Bahia de Caragues" or simply "Caragues" (Caracas). Bahia is the most important cacao of Brazil, which has increased its output of this quality from six million kilograms in 1894 to 24½ million kilograms in 1910. Bahia cacao is classed as "ordinary" and may compete with Trinidad cacao at its best, but it is more commonly priced with Samaná and San Thomé cacaos. England, France and Switzerland are large purchasers.

Beans : Of irregular contour, flat. Shell : Sometimes "clayed," if of better quality, otherwise grayish. Kernel : Red-brown to brown-violet. Flavour : Poor, bitter and smoky.

CUBA	{ 1914 output, 1,811 tons.
	{ 1917 output, 1,475 tons.

The increase in production of Cuban cacao is not great, though, up to 1914, there was a slow rise in production. The method of preparation leaves much to be desired and, though the cacao may be found listed on many of the European markets, most of it goes to Spain.

Beans : Flat and irregular, often two or three stuck  
w.c. 7



together. Shell: not easily detached from the kernel frequently found with dried portions of pulp adhering. Kernel: Dark violet in colour. Flavour: Bitter but aromatic, in which the chocolate flavour is not very pronounced after roasting.

#### Other American Cacaos.

BRITISH HONDURAS { 1914 output, 9 tons.  
1917 output, 8 tons (estimated).

Cacao is of minor importance in this colony, which produces chiefly gum chicle, mahogany, bananas and coconuts. In experiments, Forastero cacaos have shown the best growth, but there is no indication that an increase in output of cacao is to be expected.

#### NICARAGUA.

The Criollo variety of Nicaragua is very fine and larger even than Venezuela cacaos. One large plantation belongs to the well-known French chocolate manufacturer, M. Menier who grows a Forastero variety. Fermentation at this plantation is carried on for four or five days, the Criollo cacao for about two days.

COSTA RICA { 1914 output, 325 tons.  
1917 output, not known.

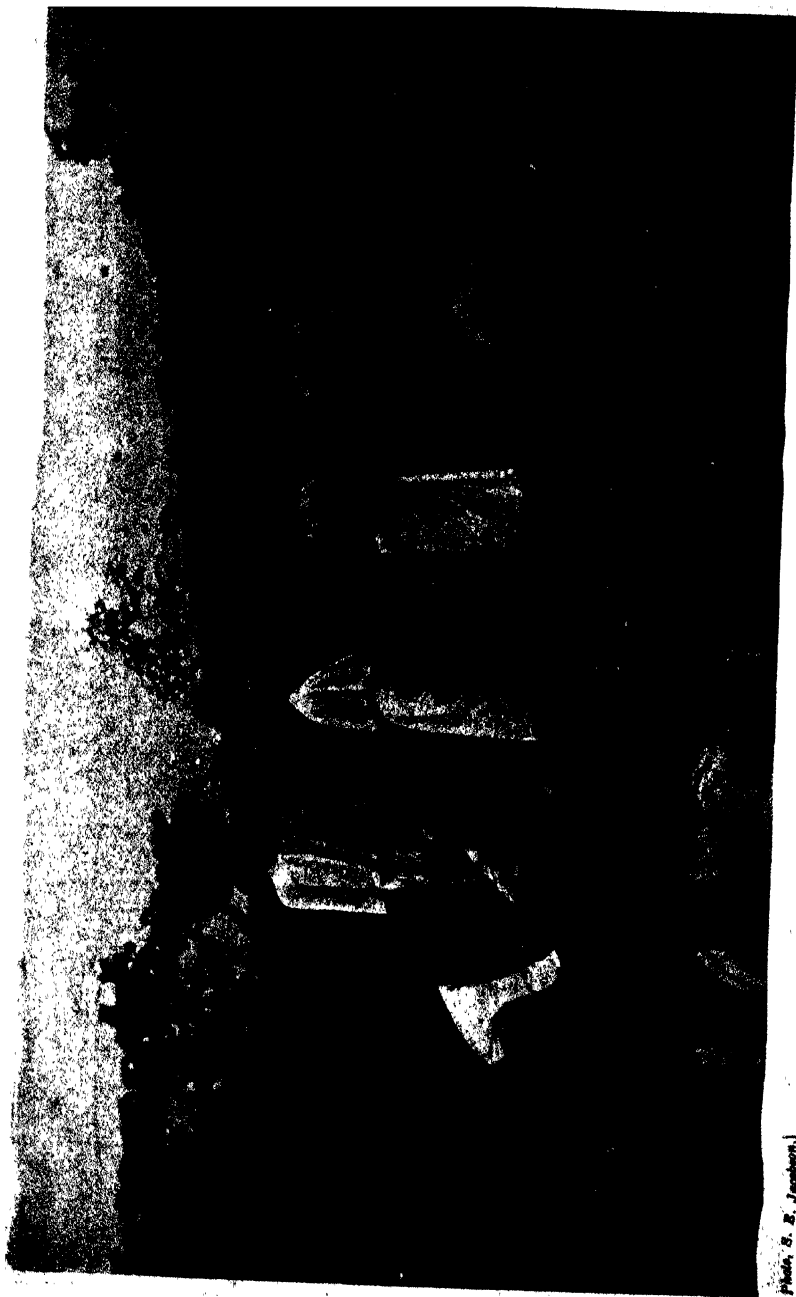
The best class of cacao is obtained from Matina near Port Limon, but the cultivation is of little importance.

#### COLOMBIA.

Approximately 400 tons annually are exported, mostly to France, the produce from the Cauca valley being best known in Europe. A considerable amount of the cacao grown, is converted into chocolate in local factories, which flourish owing to the extreme popularity of the sweetmeat among the natives.



PLATE XIV.



BAGGING CACAO FOR THE LOCAL MARKET, TRINIDAD.

Photo. G. E. Jephson.

[Trinidad]

## MEXICO AND GUATEMALA

"Royal Soconusco" has been attributed to Mexico, but, in the district of Soconusco, Mexico, very little cacao is found. The neighbourhood of Tabasco produces, annually, about one million kilograms, and Chiapas about  $1\frac{1}{2}$  million kilograms, Vera Cruz, Guerrero and other places about half million kilograms each. "Tabasco" is usually considered to be the best of the Mexican cacaos, the taste and appearance of the beans being good, and the aroma not very strong. It is owing to the lack of flavour and aroma that "Tabasco" has not met with greater popularity in Europe.

Guatemala cacao is similar to "Tabasco," of light colour and feeble aroma. The production is estimated at about 200 tons annually.

It is worthy of record that the Central American people are not partial to cacaos from abroad for their own consumption, but prefer their own local produce, each native his own country's cacao. Very little sugar is added or necessary, owing to the mild flavour of the Central American cacaos. Apart from the fact that the natives of Central America were probably the first persons to consume chocolate, it is surmised that the palate of these people is more discriminating, in that they prefer a mild, yet full-flavoured, and only slightly sweet confection to the sickly product of the European manufacturer, who is compelled to load his chocolate with sugar, in order to cover the harshness and bitterness of such cacaos as "Bahia," "San Thomé" and "Accra."

## West Indian Cacaos.

HAITI { 1914 output, 2,092 tons.  
1917 output, 1,518 tons.

Haiti was one of the earliest islands to cultivate cacao, and it is mentioned in the seventeenth century in that connection. Progress has been very slow and fluctuating,

though, in 1912, it reached a height of 3,000 tons. Like "Samaná," the Haiti cacao is of low quality, owing to careless preparation. There is little uniformity among the beans, and, often, dirty admixtures, which do not encourage popularity among the purchasers, are included. The author has been unable to obtain samples of genuine "Haiti" and can give only the appearance of the beans as recorded by others :

Beans : Smaller and of less value than those from the American continent, but somewhat similar to "Para."  
 Shell : Often very much "clayed" and, consequently, very brilliant in colour. Kernel : Dark brown or purple.  
 Flavour : Poor and unpleasant.

SAN DOMINGO 1914 output, 20,417 tons.

1917 output, 23,900 tons (estimated).

The republic of San Domingo, though a part of Haiti, has shown considerable progress in cacao cultivation. The rapidity of the growth of this industry is shown by van Hall, who gives figures of total output, in 1890, as 1,400,000 kilograms and, in 1912, as 20,900,000. The cacao is known as "Samaná" and is of the variety of smooth Amelonado, originally brought from Trinidad, though fine Venezuelan Forastero cacaos have been imported with some success. The main crop is picked in March to June, and a less important crop in October to December. Of "Samaná," there are both "plantation" and "local" qualities, the latter being the produce of the small farmers. The former commands a rather high price, though the *Gordian* classes both as "Mittelkakao," which, indeed, it is, being a little superior to that of "Accra" from the Gold Coast. Sanchez, San Domingo and Samaná, from the last-mentioned of which the cacao takes its name, are the chief ports of shipment. At present, the two first-mentioned ports ship the greatest part of the cacao for the district.

Beans : Of irregular shape, flat. Shell : Red-brown in

colour. Kernel: Blue-black on the outside, dark maroon colour towards the centre. Flavour: Bitter and poor.

TRINIDAD (1914 output, 28,325 tons.  
(1917 output, 31,315 tons.

Most uniform of all the cacaos, to which a great deal of attention has been paid. It was stated a few years back that the cacao had deteriorated in quality, and a Commission was appointed, in 1914, to inquire into the matter. It was found that the Trinidad cacao had fallen in value only relatively to some other cacaos, owing to greater care being now taken with the latter. Criollo cacao is not grown in Trinidad, but mixed varieties, all of Forastero type, make the bulk of the cacao seem uneven. The main crop is gathered in November, December, January and February. Fermentation is not yet very uniform, and the beans are either dried direct or "clayed" and "polished," as already described. Trinidad cacaos are of the finer sorts and usually figure after Ecuador.

Beans: Large, broad, more or less flat. Shell: Smooth and polished, of red-brown hue, when treated after fermentation, liable to show mould, when untreated. Kernel: Red-violet or red-brown. Flavour: Pronounced fine chocolate flavour, when properly fermented. Superior "Trinidad" comes on the market under variously branded names, of which Chaguaramas, La Réunion, L'Esperance and San Juan may be quoted.

GRENADA (1914 output, 5,135 tons.  
(1917 output, 5,000 tons (estimated).

Grenada cacao has shown very great improvement of recent years and is classed among the finer qualities. It may be compared to "Trinidad," "Surinam," and "Carupano."

Beans: Smaller than, but similar to, "Trinidad." Shell: Pale red-brown, usually loose and easily detached.

Kernel: Dark blue-violet, with pale fracture. Flavour: Slightly bitter, but similar to "Trinidad."

JAMAICA (1914 output, 3,115 tons.  
(1917 output, 3,000 tons (estimated).

The increase in the industry is slow, though of considerable importance, and is simultaneous with the increase in the banana industry. The type of cacao grown is Forastero, as in all the Antilles.

The quality is similar to "Trinidad," but slightly inferior. In the *Annual Report of the Department of Agriculture*, 1914-15, it is stated: "There is every reason to place the highest confidence in the soundness of cacao as one of the leading staples."

Beans: Usually flat and long, irregular. Shell: Better sorts similar to "unclayed Trinidad," poorer qualities have thick shells and are moist to the touch. Kernel: Brown, or purple-violet. Flavour: Only slightly aromatic, rather bitter and harsh.

#### Other West Indian Cacaos.

GUADELOUPE (1914 output, 1,106 tons.  
(1917 output, not known.

Cacao was introduced into Guadeloupe from Martinique and is mostly of Amelonado variety. Progress is not likely to be made in cacao cultivation in the island, owing to the restricted area suitable for the crop.

Beans: Similar in appearance to "Jamaica." Shell: Gray and often moist to the touch. Kernel: Usually brown, when well fermented. Flavour: Astringent and poor.

MARTINIQUE (1914 output, 440 tons.  
(1917 output, not known.

Towards the end of the eighteenth century, Martinique and San Domingo produced almost the whole of the cacao

consumed in France, and but little progress has since been made. As in Guadeloupe, Amelonado variety of cacao is mostly grown.

The product of Martinique is very similar to Jamaica cacao, with which it compares in price on the European market.

Beans : Usually long and flat. Shell : "Unclayed," often gray or mouldy. Kernel : Violet or red-brown, usually the former. Flavour : Slightly acid, vinegary.

ST. LUCIA { 1914 output, 3,615 tons.  
              { 1917 output, 3,000 tons (estimated).

\*St. Lucia cacaos are usually quoted in the London market somewhat below the best grades of "Grenada." The methods of cultivation are much the same as in Trinidad, and the product is of about the same price as the lower qualities of beans from that island. St. Lucia cacao is quoted (early 1920), "ordinary to fine," 120s. to 122s., with "Grenada" at the same price.

DOMINICA { 1914 output, 430 tons.  
              { 1917 output, 300 tons (estimated).

Dominica lies between Martinique and Guadeloupe, and there has been little or no progress made in the development of the cacao industry. The cacao is, in quality, lower than "Grenada" and is unimportant in the European markets. It is quoted to-day (early 1920) at 115s. to 130s.

ST. VINCENT { 1914 output, 100 tons.  
               { 1917 output, 59 tons.

It is probable that, in St. Vincent, cacao-growing may assume some importance in later years. Both in the Windward Islands and in the Leeward Islands there are schemes on foot for instructing students in agricultural matters. In the latter islands, Montserrat produced about 1 ton of cacao in 1914 and about 2½ tons in 1916. St. Vincent



cacaos are quoted to-day (early 1920) in the market, "ordinary to fine," 115s. to 130s.

### African Cacaos.

SAN THOMÉ (1914 output, 32,793 tons.  
(1917 output, 30,388 tons.

The island of San Thomé is about half covered with cacao plantations which produce the principal crop in October and November and a smaller crop in March and April. Fermentation is usually allowed to continue for five or six days, when the beans are dried. According to Chevalier,\* the common variety of cacao grown in San Thomé is "Creoulo," originally imported in 1822 from Bahia, in Brazil, by the Portuguese, and must not be confused with "Criollo." "Creoulo amarillo" and "C. colorado" are both grown, and another variety, called by Chevalier, *Theobroma sphaerocarpa*, and by the Portuguese "Cacao caranja" or "Carupano" or "Cacao amarillo redondo," is also to be commonly found. San Thomé cacao is classed by the *Gordian* as *Mittelkakao* and lies, in quality, between Bahia and Samaná cacaos. England, Germany, Holland and America have all been large consumers of this cacao. San Thomé was placed fourth on the list of cacao-producing countries in 1916, being preceded by the Gold Coast, Brazil and Ecuador, and fell to fifth place in 1917, Trinidad just passing it in the latter year.

Beans: Very variable, flat, rather convex on one side. Shell: Rich brown, and easily detached from the kernel. Kernel: Rich brown. Flavour: Soft and aromatic to the palate.

CAMEROON (1914 output, 4,000 tons (estimated).  
(1917 output, not known.

Of the late German African colonies, Cameroon (Kamerun) was the most important in cacao production.

\* Aug. Chevalier, "Le Cacaoyer dans l'Ouest Africain."

Samoa had about one-fourth the output of Cameroon, and Togo about one-fortieth. "Cameroon" is "ordinary" cacao about equal to "Bahia" and fair grade "San Thomé." The type of cacao grown is Forastero, but the planting of Criollo types has been considered. It is doubtful, however, whether Criollo would thrive in this rainy country. Besides "Cameroon," there is also an inferior cacao called "Victoria," which is about on a level for quality with "Accra."

Beans: Small, flat, irregular. Shell: Rough, due to imperfect artificial drying. Kernel: Dark violet, harsh, coarse and bitter to the palate, sometimes smoky, from the artificial heat used in drying.

GOLD COAST (ACCRA) 1914 output, 52,883 tons.  
1917 output, 90,964 tons.

The growth of the cacao industry in the Colony and Protectorate of the Gold Coast is phenomenal, rising from 185 tons in 1898 to nearly 91,000 tons in 1917, or a rise in sterling of some £7,500 to over £3,000,000. In 1917, the United Kingdom took over 40,000 tons, and France and America about half that quantity each. The variety of cacao grown is a Forastero-Amelonado, similar to the common San Thomé cacao, and appears on the market as "Accra." It is a mistaken notion to imagine that Gold Coast cacao is unfermented; it is, mostly, insufficiently fermented or carelessly fermented, with the result that the product is of low quality. Added to insufficient fermentation, the beans are not washed, are carelessly dried, resulting in much mouldiness, and are imperfectly selected, if at all. Progress is, however, being made in instructing the native how to secure a better product, and, within the next few years, considerable improvements may be expected.\* "Accra" is at present the lowest grade of cacao on the market, with the exception, possibly, of Haiti beans.

\* For agricultural experiments, see *Rep. Imp. Inst.*, No. 71, June, 1910.

**Beans :** Small and irregular. **Shell :** Dirty brown, often covered with mould. **Kernel :** Variously coloured, ranging from purple to brown, according to extent of fermentation. **Flavour :** Harsh and bitter.

FERNANDO PO (1914 output, 3,093 tons.  
(1917 output, 3,687 tons.

The cacao is chiefly grown in the districts on the coast, where primitive methods of curing the beans are adopted. The method of cultivation is similar to that in San Thomé. The general description of the beans is the same as for San Thomé cacao.

#### Other African Cacaos.

NIGERIA (1914 output, 4,938 tons.  
(1917 output, 15,441 tons.

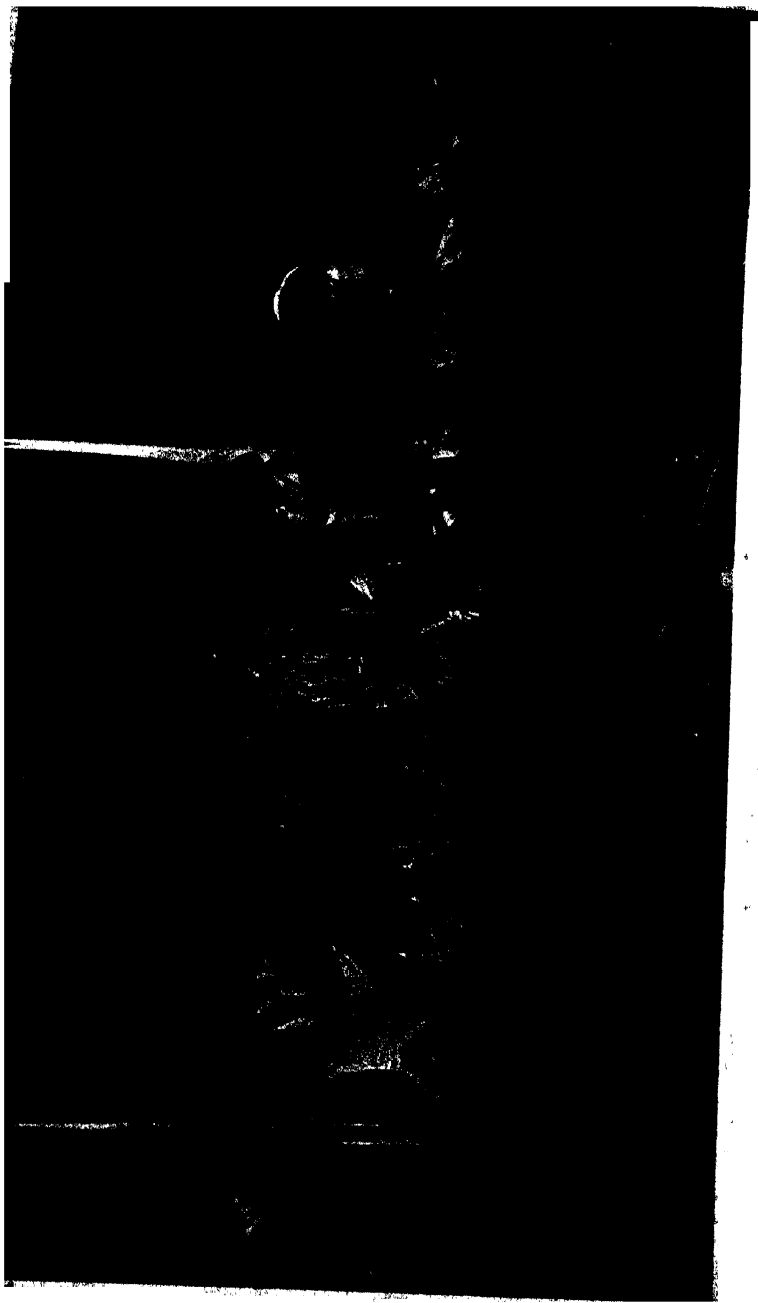
Most of the cacao exported from Nigeria is known on the European markets as "Ibadan" and is of a low grade, inferior to the ordinary Gold Coast product. The variety, usually grown, is Amelonado and is capable of great improvement. The Department of Agriculture of the Southern Provinces is encouraging scientific methods of cultivation and curing, and it is probable that improvement in the quality of the cacao will be shortly noticed.

The cacao from Lagos Colony is of a similar type and quality.

UGANDA (1914 output, not known.  
(1917 output, 15 tons (estimated).

Uganda is making only very slow progress, but is being encouraged by the establishment of experimental stations at Kampala and elsewhere. Sierra Leone also grows a small amount of cacao, but, owing to its bad climate, is not likely to become a cacao-growing colony of any importance. Much the same may be said of East African Protectorate.





BAGGING CACAO BEANS FOR SHIPMENT THIRTEEN

## CHARACTERISTICS OF COMMERCIAL CACAO BEANS 107

SAMOA {1914 output, 650 tons (estimated).  
(1917 output, not known.

Both Criollo and Forastero types of cacao are grown, the former resembling Java and Ceylon Criollo.

FIJI {1914 output, 5 tons.  
(1917 output, not known.

The production of cacao is unimportant in Fiji and is unlikely to be extended, owing to the cyclones, prevalent in the neighbourhood, and to the mountainous nature of the district.

MAURITIUS {1914 output, 1 ton.  
(1917 output, not known.

SEYCHELLES.—About  $\frac{1}{2}$  ton annually.

The cacao industry in both Mauritius and Seychelles is of little importance and dying in the latter place, where more attention is being given to growing coconuts, vanilla and rubber, and to the fisheries.

BELGIAN CONGO {1914 output, 475 tons.  
(1917 output, 765 tons.

The Belgian Congo has not yet become an important cacao-producing country. The cultivation is in the hands of small proprietors, who pay more attention to rubber than to the production of cacao.

### Asiatic Cacaos.

CEYLON {1914 output, 2,781 tons.  
(1917 output, 3,635 tons.

Ceylon Criollo or "Old Red Ceylon," famous all over the world, is gathered in October to January. The beans are fermented, and they are usually washed when a short fermentation has been adopted, plantation "Ceylon" being particularly clean. The Ceylon beans resemble the Java produce closely and are of light colour and sweet taste. The produce of Ceylon is not uniform, and there is a good deal of poor cacao

put on the market. The native "Ceylon" is usually of the lower quality, and, owing to the introduction of Forastero types, much of the cacao is of poorer flavour than the best "Old Red Ceylon."

Beans: Very pleasing appearance, smooth and round. Shell: Clean and light coloured. Kernel: Usually chocolate-brown, sometimes violet. Flavour: Mild and sweet, when properly fermented. Lower qualities often bitter or astringent.

JAVA { 1914 output, 1,555 tons.  
 { 1917 output, 1,530 tons.

Like the Ceylon Criollo, Java Criollo is of very fine quality. This variety is usually fermented for one to three days, and every day the cacao is turned over at least once. The Forastero variety is fermented a little longer, and the beans are nearly always washed. As in Ceylon, the beans are dried in the sun, whenever possible, and sorting is carefully carried out. The result is an excellent product, generally uniform in quality. All the Java cacao is shipped to Amsterdam and sold at public auction from each plantation separately. "Widodaren" and "Getas" are the names of two of these plantations. There is some poor quality "Java" put on the market, especially that infested by the "cacao moth," which attacks the pods and the beans before picking, and which causes enormous losses annually.

Beans: Rather larger and rounder than the best "Ceylon." Shell: Slightly lighter brown than Ceylon cacao. Kernel: Pale brown. Flavour: Mild and somewhat aromatic.

#### Other Asiatic Cacaos.

The other islands of the Archipelago, besides Java, that produce cacao are Celebes (Makassar and Menado), Ternate, Amboina, Sumatra (West and East) and Bali, to a total of some 60 tons annually.

**Judging Cacao for Cocoa and Chocolate Making.**

*"De gustibus non est disputandum."* The British like "Trinidad," the Central Americans prefer their own local product, and each is an instance of insularity or conservatism. There is, however, no doubt that, in order to obtain continuously a uniform cocoa or chocolate, blending is necessary, owing to the natural variations that occur in the beans and to the variable methods of curing, which have not yet been standardised in every cacao-producing country on a scientific basis. "Value for money" is, of course, the principle that rules the market price, and value is based upon the aromatic qualities of the kernel, the actual "return" possible of kernel or "nibs" from a bulk of beans, and certain other qualifications, such as percentages of fat and theobromine.

The best cacao is obtained from the best stock, Criollo, Forastero or Calabacillo, in the order named, and must be fermented correctly, *i.e.*, on a scientific plan, and must be washed and dried sufficiently, in order to bring the beans on to the market in good condition, without adhering pulp or earth and without mould.

So far as the exteriors of the beans are concerned, they should be cinnamon-coloured, and not black or gray, mouldy or patchy. However, many marks cannot be judged on their outside appearance, as often little or no attention is paid to this aspect in their preparation, the colour and aroma of the "nibs" being the chief points of importance. Again, as in Trinidad, the beans may be "clayed" and "polished," or "clayed" only, as in some Venezuelan cacaos, processes which, whilst adding to the attractiveness of the appearance, and, in most particulars, protecting the beans from deterioration by damp, etc., are open to objections, in that they add weight to the husk, a refuse or by-product in the process of chocolate and cocoa manufacture. Losses due to husk (and, consequently, to any weight of earth attached) are considered later in tables of analyses.



With regard to the interior of the beans, the kernels or "nibs" should part easily from their shells or husks on crushing between the fingers. The whole bean should be crisp or brittle and should break down easily, the interior and exterior separating readily. Unfermented cacao does not behave in this way, and the kernel is deep violet or purple in colour, with a sour flavour, and is difficult to separate from its shell.

As has been already observed, fermentation modifies both the colour, from violet to chocolate-brown, and the tannins and other astringent matters by oxidation to less objectionable substances. Consequently, an imperfectly fermented bean will be both violet in colour and bitter or astringent to taste. Sometimes, if the beans have been allowed too much fermentation or are improperly washed and dried, a distinct sour, vinegary odour will be noticed which, whilst being largely removed in the subsequent roasting, is indicative that care has not been expended in their curing on the plantations.

Another item to be considered in judging cacao is the number of beans going to a given weight, or the weight of a given number of beans, the heavier the beans the higher the price, all other things being equal.

Thus one record\* is shown as follows:—

Cacao.	Weight of 100 beans.
Trinidad (ordinary) . . .	98 grammes.
Trinidad (good). . .	132.2 „
Trinidad (fine) . . .	178.7 „
Grenada (medium) . . .	104.5 „
Grenada (fine) . . .	131.0 „
Caracas (good) . . .	130.3 „
San Domingo (good) . . .	110.0 „
Surinam (fine) . . .	122.0 „
Surinam (small beans) . . .	71.5 „

\* "Fabrication du Chocolat," 1910, by J. Fritsch.

Bahia (good)	. . .	118.0 grammes.
Mexico (good)	. . .	136.5 ..
Africa (good)	. . .	128.0 ..

Or, again, some comparative figures from the author's laboratory show the following :—

Cacao.	No of beans weighing 4 ozs.	Loss on roasting Per cent.	Price (early 1920).
Grenada	. . 120	. 5.7	. 126s. to 127s.
Grenada	. . 116	. 5.9	. 126s. to 127s.
Grenada	. . 126	. 6.2	. 126s. to 127s.
Trinidad.	. . 94	. 7.1	. 123s.
Venezuela (fine)	. . 72	. 6.25	. 167s. 6d.
Accra	. . 116	. 5.35	. 112s.

In these last cases, it will be observed that size had little or nothing to do with the market price, but, as will be seen from the column under "Loss on Roasting," the Trinidad beans were more dirty or moist than the Grenada cacao and were, consequently, of an inferior grade.

The amount of total ash is indicative also, to some extent, of the cleanliness shown in preparation of the cacao, or of the amount of earth left on the shell. The following table is by Fritsch :—

Cacao	Ash per cent. of the whole bean.
Trinidad (ordinary)	. . . 3.37
Trinidad (extra fine)	. . . 3.62
Trinidad (good)	. . . 3.64
Mexico	. . . 4.27
San Domingo	. . . 2.82
Grenada (fine)	. . . 3.12
Grenada (medium)	. . . 3.06
Caracas	. . . 4.58
Bahia	. . . 3.31
Africa	. . . 2.68
Surinam (fine)	. . . 3.06
Surinam (small beans).	. . . 3.15

The value of washing cacaos is shewn in the following table, taken from a Report of The Imperial Institute (June, 1910) :—

Method of preparation	Husk Per cent	Calculated on the husked samples. Per cent.			
		Moisture.	Fat	Ash	Total alkaloid
I. Fermented 8.5 days, and washed	8.0	4.55	48.29	2.39	1.28
IVa. Fermented 4.5 days, and washed.	8.0	4.87	46.63	3.05	1.65
IVb. Fermented 4.5 days, unwashed.	8.0	4.75	46.17	2.90	1.58
Va. Fermented 6.5 days, and washed	8.0	4.89	44.51	2.74	1.20
Vb. Fermented 6.5 days, unwashed.	11.4	5.00	45.30	2.66	1.40
VIa. Fermented 7.5 days, and washed.	8.4	4.55	44.50	2.67	1.22
VIb. Fermented 7.5 days, unwashed.	10.4	4.90	45.20	2.87	1.21

TABLE XI.—*Proportion of Nibs, Husk and Germ in some Cacaos.*

Cacao	Number of beans per lb	After roasting				Loss on roasting. Per cent.
		Nibs Per cent.	Husk Per cent	Germ Per cent	Fat in nibs. Per cent.	
1. Trinidad	380	85.50	13.74	0.61	—	6.25
2. "	288	83.06	15.65	0.70	53.62	7.14
3. "	440	87.96	11.23	0.81	52.83	—
4. "	376	87.32	11.05	0.73	49.78	7.10
5. " (San Juan)	336	84.51	14.64	0.67	52.74	2.08
6. Grenada	396	86.82	12.40	0.78	55.70	—
7. "	432	82.06	13.49	0.82	59.20	4.46
8. "	420	84.83	14.16	0.91	46.00	4.46
9. "	444	86.25	12.49	0.80	54.98	5.36
10. "	420	87.02	12.16	0.71	56.23	5.35
11. "	384	87.15	11.97	0.75	53.94	5.35
12. "	428	85.18	13.90	0.67	49.40	5.36
13. "	424	83.48	15.38	1.02	50.92	3.57
14. St. Lucia	428	86.30	12.94	0.67	52.52	3.57
15. "	392	85.79	13.29	0.76	50.90	3.57
16. "	412	86.19	13.07	0.73	54.16	5.80
17. "	364	85.96	12.81	0.78	58.10	4.46
18. "	388	86.14	15.00	0.71	55.24	3.57
19. Puerto Cabello	352	85.18	13.50	0.79	53.64	1.70
20. Venezuela	476	78.00	20.10	0.92	51.78	6.25
21. " (Fine)	288	86.05	13.15	0.73	52.90	6.25
22. Acra	404	89.31	9.77	0.90	52.36	5.35

Finally, the variations shown by the same class of beans in the "return of nibs," "number of beans to the pound,"

and "loss on roasting," can be seen in the figures in Table XI., selected from many prepared in the author's laboratory in 1920.

Ridenour, in 1895, showed some interesting figures somewhat on the same lines, though this experimenter made complete analyses, some of which are recorded later. Of these cacaos, mentioned in the next chapter and analysed by Ridenour, the following are the average weights of fifty of the beans :—

Bahia, 0.856 grammes ; Surinam, 1.175 grammes ; Java, 0.994 grammes ; Grenada, 0.920 grammes ; Arriba, 1.434 grammes ; Maracaibo, 1.364 grammes ; Caracas, 1.214 grammes ; Trinidad, 1.189 grammes.

Yet another worker, Brayning,\* has recorded the average weight of beans and the proportion of shell to kernel in a number of cacaos, thus :—

Cacao	Kernel Per cent	Shell. Per cent.	Mean weight of unshelled beans. Grammes.
Java . . . . .	92.9	7.1	1.236
San Thomé . . . . .	92.3	7.7	1.348
Surinam (1) . . . . .	91.4	8.6	1.149
Trinidad . . . . .	90.9	9.1	1.286
Para . . . . .	89.8	10.2	1.136
Porto Plata . . . . .	89.5	10.5	1.292
Haiti . . . . .	88.6	11.4	1.317
Bahia . . . . .	88.4	11.6	1.379
Puerto Cabello . . . . .	88.1	11.9	1.598
Surinam (2) . . . . .	88.1	11.9	1.637
Guayaquil (Machala) . . . . .	88.0	12.0	1.537
Guayaquil (Arriba) . . . . .	87.0	13.0	1.628
Carupano . . . . .	86.8	13.2	1.469
Caracas . . . . .	86.6	13.4	1.504
Grenada . . . . .	86.6	13.4	1.290

The following descriptions of different varieties of beans, recorded by the author at the commencement of 1909,

\* Brayning, *Jour. d'Agric. Tropicale*, June, 1901, 31.

compared with the market prices at that time and with those at present ruling, give some indication of the relation that exists between physical characteristics of the beans and their value to the chocolate manufacturer.

*Ordinary Jamaica* (50s., 1909; 118s., 1920).—Bean: Irregular, damp to the touch, strong vinegary and winy odour. Shell: Damp, not easily detachable, tough, sometimes covered with white efflorescence, 11·1 per cent. of the bean. Kernel: Harsh and bitter, pale violet to red-brown, usually showing white at fracture.

*Native Ceylon* (51s., 1909; 100s., 1920).—Bean: Medium, irregular but flat. Shell: Thin, dirty, not easily detached, 10·75 per cent. of the bean. Kernel: Dull red-brown, bitter but pleasant flavour.

*San Thomé* (55s., 1909; 131s., 1920).—Bean: Small, dry, clean, shiny, flat. Shell: Cinnamon-brown, thin, dry, showing veins, easily detachable, 11·07 per cent. of bean. Kernel: Violet-brown, pleasant aromatic chocolate flavour, dry and brittle. A good, cheap bean in 1909.

*Superior Bahia* (57s., 1909; 130s., 1920).—Bean: Small, irregular, showing veins. Shell: Varying considerably in cleanliness, dry, thin, readily detachable, 11·3 per cent. of the bean. Kernel: Red-brown, uninteresting, bitter.

*Middling-red Trinidad* (62s., 1909; 137s., 1920).—Bean: Irregular, hard, clean, flat. Shell: Red-brown, thin, tough, not readily detached, 12·5 per cent. of bean. Kernel: Tough, rich violet or violet-brown, pleasant cacao flavour.

*Fine Guayaquil Arriba* (72s., 1909; 170s., 1920).—Bean: Irregular, rough, flat, dry pulp often adhering and causing beans to stick together. Shell: Red-brown to nearly black, dry, not easily detached, 11·2 per cent. of bean. Kernel: Rich dark brown, fine scented flavour, dry and friable.

*Finest Trinidad* (70s., 1909; 146s., 1920).—Bean: Medium, irregular, slightly flattened. Shell: Thin, dry,

showing veins, not easily detached, 15.9 per cent. of bean. Kernel: Brown to violet, fine cacao flavour, slightly bitter.

*Good Plantation Ceylon* (77s., 1909; 200s., 1920).—Bean: Plump, olive-shaped. Shell: Thicker than "Native Ceylon," brick-red, very clean, dry, easily detached, 7.4 per cent. of bean. Kernel: Brown, fine, pleasant aromatic flavour, soft and fatty.

*Good Middling Costa Rica* (56s., 1909).—Bean: Clean, hard, dry, shiny, flat on one side. Shell: Dry, thin, easily detachable after the bean is broken, 10.5 per cent. of the bean. Kernel: Friable, slightly bitter and astringent, devoid of pronounced flavour.

Some figures, given by Hart, on the valuation of cacao are interesting. He showed three samples: I. "Venezuelan Fine Clayed," valued at 14 cents. per pound; II. "Trinidad Fine Estates," valued at 14½ cents. per pound; and III. "Trinidad Ordinary," at 14 cents. per pound, and he estimated the losses during roasting, etc., in much the same way as is performed in the author's laboratory. The following are Hart's results:—

TABLE XII.—*Losses in Manufacture of Cocoa Powders.*

		I. Venezuelan. 14 cents. per lb.	II. Trinidad. 14½ cents. per lb.	III. Trinidad 14 cents. per lb.
		Lbs.	Lbs.	Lbs.
1. Weight received . . . . .		10	10	10
2. When roasted and cleaned . . . . .		7.55	7.86	7.80
3. Weight of husk . . . . .		1.68	1.63	1.53
4. Weight of dry cacao after fat was removed . . . . .		5.50	5.60	5.48
5. Weight of fat extracted from (2) . . . . .		1.23	1.64	1.61
6. Loss during roasting and cleaning . . . . .		0.77	0.51	0.67
7. Loss during grinding and expressing of fat . . . . .		0.82	0.62	0.71
8. Total loss in manufacture . . . . .		1.59	1.13	1.38
		Per cent.	Per cent.	Per cent.
10 lbs. { Dry cacao powder . . . . .		55.0	56.0	54.8
{ Fat . . . . .		12.3	16.4	16.1
{ Loss . . . . .		32.7	27.6	29.1
{ Husk . . . . .		—	11.3	13.8
			16.3	15.3

The losses, shown, are certainly greater than those occurring in bulk, and it will be noticed that the clay, when comparing samples II. and III., is estimated at about  $1\frac{1}{2}$  per cent.

TABLE XIII.—*Average Weights and Dimensions of Fresh and Cured Beans.*

Cacao.	Average for cured beans.			Average weight of cured beans.	Average weight of fresh beans.
	Length.	Breadth.	Thickness.		
	In millimetres.	In millimetres.	In millimetres.		
Arriba . . . . .	24	15	6	Grms. 1.75	Grms. —
Machala . . . . .	22	13	5	1.17	—
Bahia . . . . .	23	14	4	1.20	—
Trinidad . . . . .	25	18	4	1.29	—
Caracas . . . . .	23	15	8	1.77	—
Puerto Cabello . . . . .	24	15	8	1.25	—
Peylon . . . . .	20	12	7	1.00	—
Java . . . . .	23	12	9	0.80	—
Variety					
Forastero Liso Colorado . . . . .	25.88	15.12	10.83	1.06	3.89
Forastero Liso Amarillo . . . . .	25.53	14.37	9.98	1.34	3.34
Forastero Amelonado pequeño . . . . .	24.78	14.13	8.40	0.93	2.11
Forastero amelonado . . . . .	25.18	14.82	8.83	1.29	2.80
Theobroma sphaerocarpa . . . . .	21.29	15.02	9.13	1.21	2.54

The figures, shown in Table XIII., are not, of course, in any way absolute, but merely indicative of the average size and weight of the different varieties.

To recapitulate briefly—a well-fermented bean is apparent by its brittleness and the ease with which the shell is removed from its kernel, and from the brown colour (with absence of violet) of the interior, from its mild flavour and absence of bitterness and astringency. This applies to the better varieties of cacao grown, and, clearly, the characteristics must alter somewhat for different districts.

“Caracas,” “Puerto Cabello” and “Maracaibo” are often covered with light red or brick-red earth, “Trinidad”

being often the same, only with smooth polished surface instead of "matt." Java and Ceylon cacaos are usually very clean. Ecuador cacaos are very uneven in colour and appearance, but the finer sorts are of very high quality, and they command high prices. The finer qualities of Venezuela (Western), Ceylon and Java cacaos are pale brown inside and of very mild and somewhat sweet flavour.

Generally, the darker the kernels the more bitter the flavour, and, though many of the finest sorts, such as "Caracas," "Arriba," "Puerto Cabello," etc., have a pronounced chocolate flavour even before roasting, in most cacaos the aroma is hidden by the vinegary smell (acetic acid), developed during fermentation.

#### **Cacao Tasting.**

Schulte im Hofe recommends the testing of cacao by taste as well as by observation of colour and aroma. Fifty grammes of the beans are roasted, shelled and cooled. The nibs are crushed in a pestle and mortar or small mill and rubbed down to a fine powder, of which 6 grammes are added to 100 ccs. of water and brought to the boil. H. H. Smith recommends the use of half water and half milk, to replace the water. The infusion is judged for flavour, aroma and colour.

The present author uses a small coffee roaster, holding 1 lb. of beans, and has been able to get consistent roasts with a little practice. As seen from the foregoing tables, the samples are judged for "refuse" (shell and germ) and "loss on roasting," besides colour, aroma and flavour.

The development of the aromatic oils, which takes place during the roasting of cacao, just as during the roasting of coffee, is of the greatest importance to the manufacturer who considers the flavour of his cocoa and chocolate, and must, consequently, be taken into account when a variety of bean is purchased.



The percentage of moisture and fat, both of great economic importance to the manufacturer, cannot be accurately gauged by consideration of the external qualifications of the bean, alone, or without aid of the chemist. It is proposed, therefore, in the next chapter, to give the chemical composition of the commercial bean in general and of the varieties in particular, found by the author and by other workers, among whom should be mentioned Ridenour, Harrison, Zipperer, Dekker, Booth and Ewell.

## CHAPTER IX

### COMPOSITION OF CACAO BEANS

CACAO beans, in common with other seeds, contain all the substances necessary to nourish the growing plant during germination, and, apart from water, the component parts may be divided up under the headings "organic" and "inorganic" constituents.

The organic bodies may, in turn, be divided as follows :—

(1) Carbohydrates—such as starch, cellulose, lignin, saccharose [sucrose or cane-sugar], glucose.

(2) Oils and fats—consisting in the main of olein, palmitin, stearin, and their mixtures.

(3) Pectose group—pectose, pectin and pectic acid.

(4) Vegetable acids—such as tartaric and acetic acids.

(5) Albuminoids or proteids—such as vegetable albumen.

(6) Amides—diffusible nitrogenous bodies, such as asparagin.

(7) Extractives—such as chlorophyll, tannin, and the alkaloids.

The inorganic constituents consist of salts of various metals, which occur in plant life mainly as :—

Inorganic acids	Phosphates	of	Potassium.
	Nitrates		Sodium.
	Silicates		Calcium.
	Sulphates		Magnesium.
	Chlorides		Iron.
	Carbonates		Manganese.
Organic acids	Oxalates		Aluminium.
	Malates		
	Tartrates		
	Acetates		
	Citrates		

TABLE XIV.—*Analyses of Raw Beans of "Calabacillo" Variety, grown in Trinidad.*

	Beans, cuticles and pulp.			Cuticles		Kernel.		
	Fresh	Dried.	Fermented and cured.	Dried.	Fermented and cured.	Fresh.	Dried.	Fermented and cured.
Water	61.780	5.000	7.169	12.400	12.400	37.637	5.000	6.080
Albuminoids	3.904	0.704	7.213	6.002	6.750	6.696	10.202	7.310
Indeterminate nitrogenous matter	0.274	0.681	3.509	Traces.	4.006	0.531	0.809	3.406
Theobromine	0.814	2.023	1.549	1.599	1.023	1.352	2.059	1.659
Caffeine	0.075	0.186	0.103	0.272	0.355	0.108	0.164	0.058
Fat	15.361	38.181	40.744	2.046	4.000	20.256	44.574	48.400
Glucose	0.862	2.143	0.900	4.811	0.476	0.091	1.510	1.000
Sucrose	0.032	0.070	0.024	0.240	0.143	Traces	Traces	Nil.
Starch	2.406	5.980	5.249	6.271	4.865	3.764	5.735	5.320
Astringent matter	2.776	0.900	5.306	2.621	2.113	5.004	7.624	5.072
Pectin, etc	0.773	1.822	2.671	5.408	6.140	0.657	1.386	1.650
Cacao-red	1.772	4.404	2.420	3.391	3.900	2.952	4.497	2.300
Digestible fibre, etc	4.847	12.048	11.815	36.388	35.721	5.112	7.287	6.182
Woody fibre	2.219	5.515	5.503	8.932	9.840	3.030	4.617	4.600
Tartaric acid (free)	0.253	0.620	0.535	2.913	0.420	0.070	0.120	0.560
Acetic acid (free)	Nil.	Nil.	0.869	Nil.	0.720	Nil.	Nil.	0.900
Tartaric acid (combined)	0.192	0.074	1.114	2.019	3.450	0.477	0.726	0.624
Iron peroxide	0.018	0.044	0.105	0.026	0.057	0.042	0.048	0.115
Magnesia	0.225	0.550	0.686	0.756	0.990	0.324	0.493	0.621
Lime	0.054	0.134	0.207	0.358	0.266	0.054	0.082	0.106
Potash	0.528	1.312	1.125	1.260	1.821	0.142	1.283	0.980
Soda	0.143	0.355	0.120	0.272	0.219	0.239	0.364	0.477
Silica	0.060	0.022	0.065	0.013	0.200	0.016	0.024	0.037
Sulphuric anhydride	0.104	0.482	0.057	0.130	0.085	0.079	0.120	0.051
Phosphoric anhydride	0.442	1.008	1.113	0.763	0.912	0.749	1.141	1.179
Chlorine	0.012	0.044	0.020	0.119	0.019	0.019	0.028	0.021
	100.132	100.319	100.000	100.000	100.000	100.000	100.000	100.000
Total nitrogen	-	1.542	2.134	1.542	2.134	1.603	2.472	2.240

In cacao beans, the most important organic and inorganic compounds occurring are water, starch, fat, glucose, albumen, theobromine, cacao-red, cellulose or fibre, a small quantity of cane-sugar and mineral or ash, which last consists of silicates, phosphates, sulphates, chlorides, and carbonates of potassium, sodium, calcium, magnesium, iron and aluminium.

The foregoing table, which the author has arranged from a collection of strictly comparable analyses, made by Professor Harrison, throws more light on the composition of cacao beans than any number of words. This remarkable set of analyses was conducted with the intention of ascertaining the changes taking place in the cacao bean during fermentation, but, in the general way, it is quite unnecessary to carry out any such elaborate analysis.

An analysis of San Thomé cacao made in January, 1910, showed the following to be the composition of the husks and kernels :—

	Husks.	Kernels.
Moisture . . . . . per cent.	12.15	7.80
Ash . . . . .	7.25	3.72
Nitrogenous constituents . . . . .	15.70	13.73
Fat . . . . .	2.89	50.00
Theobromine . . . . .	0.62	1.37
Starch . . . . .	—	6.25
Cellulose . . . . .	15.93	3.13

The following analyses of raw commercial cacaos, after removal of husk, by Eastes and Terry,\* are more typical of general requirements :—

TABLE XV.—Analyses of Kernels of Raw Cacao.

Kind of cacao.	Moisture.	Fat.	Theo- bromine.	Ash.	H <sub>2</sub> PO <sub>4</sub> .
Caracas . . . . .	4.75	53.65	1.08	2.76	1.36
Carupano . . . . .	5.04	47.38	0.87	3.09	1.39
Grenada . . . . .	5.59	47.12	1.42	2.81	0.91
Guayaquil . . . . .	3.68	52.97	1.74	3.28	0.85
Para . . . . .	4.39	57.07	1.00	3.09	1.30
Surinam . . . . .	2.55	53.70	1.42	2.44	0.85
Trinidad (Common) . . . . .	5.62	45.71	1.05	2.79	0.89
Trinidad (St. Antonio) . . . . .	4.72	53.57	1.94	2.70	1.15

TABLE XVI.—Analyses of Kernels of Raw Cacao.

	Bahia.	Surinam	Java	Grenada.	Guaya- quil Arriba.	Surinam (Mara- calibo).	Caracas.	Trinidad.
Fat . . . . .	42.10	41.03	45.50	44.11	43.31	42.20	30.81	43.66
Theobromine . . . . .	1.08	0.93	1.16	0.75	0.86	1.03	1.13	0.85
Albuminoids . . . . .	7.50	10.54	9.25	9.76	10.14	11.56	10.59	11.90
Glucose . . . . .	1.07	1.27	1.23	1.81	0.42	1.09	2.76	1.38
Sucrose . . . . .	0.51	0.35	0.51	0.55	1.58	1.36	1.56	0.32
Starch . . . . .	7.53	3.61	5.17	6.27	6.37	1.69	3.81	4.98
Lignin . . . . .	7.86	3.90	6.10	5.55	4.62	7.16	3.28	5.65
Cellulose . . . . .	13.80	16.24	13.85	13.49	14.07	17.32	16.35	13.01
Extractives by difference . . . . .	8.99	13.53	8.90	9.72	9.00	6.79	12.72	8.31
Moisture . . . . .	5.96	5.55	5.12	5.28	5.90	5.67	6.63	6.34
Ash . . . . .	3.60	3.05	3.31	2.71	8.73	4.13	4.36	3.60

\* Eastes and Terry, *Pharm. Jour.* (3), xv., 764.

A very large number of analyses of commercial cacaos have been made by Ridenour,\* a few of which are given in Table XVI., and represent the composition of the husked bean.

Filsinger† pointed out that these figures of Ridenour could not be taken as representing normal beans, but, with the exception of the low percentages of starch in the beans from Surinam, and the rather low fat values, there are no important differences in the figures from those obtained by other observers.

The next table shows analyses of the husked bean, made by various investigators at widely differing times:—

TABLE XVII.—*Analyses of Kernels of Raw Cacao.*

	Guayaquil					Caracas					Trinidad.					Surinam.					Gulana Forn- tero				
	I	II	III	IV	V	VI	VII	VIII	IX	X	XI	XII													
Water	5.60	3.08	0.33	4.75	6.50	6.20	5.62	6.67	5.23	7.07	2.55	6.30													
Albumen	14.30	11.56	16.84	15.36	16.84	15.36	14.38	13.26	21.11	—	—	6.10													
Theobromine	1.20	1.74	0.33	1.08	0.77	0.40	1.05	0.84	0.50	1.42	1.70	—													
Fat	45.40	52.07	52.68	53.65	50.31	51.57	45.71	54.60	50.44	50.86	53.70	52.10													
Starch	14.30	—	8.29	7.65	11.07	—	—	—	4.20	6.41	—	6.80													
Woody fibre (cellulose, etc.)	5.80	—	2.89	—	3.00	3.07	—	2.45	6.40	3.02	—	18.00													
Ash	3.50	3.28	4.11	2.76	4.17	2.87	2.70	2.87	2.75	2.72	2.44	1.80													

I. Mitscherlich, "Der Kakao und die Schokolade," 1859, 57.

II., IV., VII., XI., Eastes and Terry, *Pharm. Jour.*, 1885, xv, 764.

III., V., VI., X., Zipperer, "Untersuch. über Kakao, etc.," 56, 57.

VIII. N. P. Booth, *The Analyst*, 1900, xxiv, 137.

IX. Bell, "Thorpe's Diet. App. Chem.," 1905.

XII. J. B. Harrison in "Cacao," by J. H. Hart, 1911.

Any variations which may be observed in the figures of analyses, made by different individuals, may be attributed to the varying quality of the cacaos examined, which, as has been previously pointed out, is largely dependent upon the conditions under which it has been grown and upon the processes to which the beans have been subjected. The estimation of fibre, cellulose, etc., will always differ with different

\* Ridenour, *Amer. Jour. Pharm.*, 1893, lxxvii, 202, etc.

† Dr. Filsinger, *Chem. Zeit.*, March, 1897, xxii.

observers, owing to the various methods employed in their determination. This question has been considered fully later.

As will be seen from the figures given, the principal components of cacao bean are the fat, albuminoids, starch, woody fibre, water, ash and theobromine, which together constitute about 85 per cent. of the husked bean. Of these, the fat and theobromine are of the greatest economic value, as the former provides the cacao butter of commerce, which, besides being used for chocolate-making, has certain applications in pharmacy, while to the latter are due the stimulating properties of cocoa and chocolate. Cacao-red and the aromatic oils are of far greater importance than their quantities present in cacao beans would suggest, and, though it is not usual to estimate them by analysis, the manufacturer is fully alive to their existence and importance.

Colour and aroma of the resulting cacao preparations are points which must be studied, if high-class productions are to be made. The attainment of both qualities is mainly in the hands of the planter, but the manufacturer can work wonders by careful manipulation in roasting and other processes of manufacture.

It is not intended to give, here, details of the various components which go to make up the cacao bean, these being reserved for the third part of the book, which deals essentially with the analysis and chemistry of cacao.

The results of analyses of the kernel of raw cacao have been shown, and there remains to be considered the composition of the husks or shells, which should not be employed in the preparation of cocoa and chocolate, but which should constitute a by-product of the industry.

The husks or shells do not find any application of commercial value until after the beans have been roasted, when they may be employed as cattle food, for the preparation of brown colouring or flavouring matters, for extraction of the 3 or 4 per cent. fat, or even, when crystallised in sugar,

as a sweetmeat. "Cocoa teas," made from cacao shell, have appeared again, recently, on the market, and the extraction of alkaloids from the husk has assumed some importance.

In the table of analyses by Harrison, given at the commencement of the chapter, will be found the composition of the husks of the same variety of bean, dried and fermented and cured. Other analyses show the following to be the components of raw husks :—

TABLE XVIII.—*Analyses of Husks of Raw Cacao.*

	Caracas		Trinidad.		Puerto Cabello.	
	I.	II.	I.	II.	I.	II.
Moisture . . . .	7.74	11.90	8.30	13.00	6.40	12.04
Fat . . . . .	5.90	4.15	4.23	4.74	4.38	4.00
Albuminoids . . .	11.68	13.95	15.14	13.21	13.75	—
Woody fibre . . .	12.79	17.99	18.00	18.04	14.83	15.98
Ash * . . . .	8.32	16.73	7.06	7.78	6.06	8.99

TABLE XIX.—*Analyses of Husks of Raw, Unnamed Cacao, or Mean Analyses.*

	I.	II.	III.	IV.
Moisture . . . .	7.83	12.51	—	12.40
Fat . . . . .	6.38	4.23	3.60	4.00
Albuminoids . . .	14.20	13.58	12.80	10.76
Woody fibre . . .	14.60	16.71	—	9.84
Ash . . . . .	7.12	10.20	5.70	4.58
Theobromine . . .	—	0.33	0.39	1.02

I. Laube and Aldendorff in König's *Die mensch. Nahr. u. Genussm.*, i., 201.

II. Zipperer, "Untersuch. über Kakao, etc."

III. Dekker, *Chem. Centr.*, 1902, ii., 1217.

IV. Harrison in Hart's "Cacao," 95.

With the exception of the percentages of fat in the kernel and of husk in the whole bean, the analysis of cacao previous to its roasting is of comparatively little importance and of

only academic interest. After roasting, however, the more physical properties of the nibs—such as the aroma, their capacity for readily mixing with a large amount of sugar, which is largely dependent upon the percentage of fat, the ease with which the husk is separated from the nib, and the percentage of loss entailed by this process, depending on the proportion of husk in the original bean—are of considerable moment to the manufacturer.

Analyses of the cacao germ have been made from time to time. The germ, like the husk, should be a by-product in cocoa and chocolate manufacture, owing to its poor flavour and to the grittiness which makes it difficult to refine to the smoothness necessary for the best preparations.

Haussler \* gives the following analysis, the figures being calculated in percentages of the dry substances and shown in column I. The second column shows the results of Richards,† calculated on a similar basis to Haussler's figures :—

TABLE XX.—*Analyses of Germ of Cacao.*

	I.	II.
Total ash . . . . .	6.53	7.03
Fat . . . . .	7.80	3.85
Total nitrogen . . . . .	5.16	5.29
Crude fibre . . . . .	5.26	3.51
Acidity (as tartaric acid) . . . . .	4.47	—
Dextrose . . . . .	0.44	—
Sucrose . . . . .	2.13	—
Theobromine . . . . .	1.88	—
Caffeine . . . . .	0.21	—
Total water-soluble extract . . . . .	23.40	27.63
Soluble ash . . . . .	6.26	3.85
Alkalinity of soluble ash as $K_2CO_3$ . . . . .	1.72	2.37
Iodine value of fat . . . . .	48.1	—
Acid value of fat . . . . .	19.2	—
Zeiss Butyro-refractometer value of fat at 35° C. . . . .	—	66 degrees.

\* E. P. Haussler, *Arch. Pharm.*, 1914, coln., 82 to 89.

† P. A. Ellis Richards, *The Analyst*, 1918, xliii., 214.





**PART II**  
**MANUFACTURE OF CHOCOLATES AND**  
**COCOA POWDERS**



## CHAPTER X

### CLEANING, SORTING AND GRADING CACAO, PRIOR TO ROASTING

THE cacao beans of commerce appear on the market in sacks and, according to the care with which they have been tended during the processes of fermenting and curing, are in a more or less fit state for immediate roasting.

As a rule, however, there are found, admixed with the beans, pieces of refuse matter—sticks, wood, stones, and pieces of sacking, etc.—which necessitate at least a simple sorting for their removal, and, except in the cases of the finest grades, the beans are of such various sizes that it is absolutely essential that some system of separation be employed.

Before roasting, the beans must be graded according to size, and, if the blends of cacaos, employed, show great variations, each brand of bean should be graded and roasted separately before blending. Though the need of this is obvious, seeing that small beans roast more quickly than large, resulting in an uneven roast, the importance of grading is often overlooked.

Whether the blend is to be roasted partially or fully (depending upon subsequent treatment of the chocolate or cocoa), an even roast is necessary, if good products are to result, but the need is less urgent in the former than in the latter case, since the development of flavour by heat may be conducted in the cacao mass from the nibs of partially roasted beans in later processes. In some marks of cacao, even of good quality, beans are found adhering together by pieces of dried pulp, and lumps of beans of this kind will,

clearly, roast unequally. Grading also allows a sorting-out of extraneous objects and refuse, frequently found among the beans, which cause depreciation of flavour and aroma developed during the roasting. Wood, pieces of sacking, etc., char in the roaster and are apt to impart an unpleasant empyreumatic flavour which would impair the delicacy of the aroma of the cacao mass. Pieces of iron and steel, nails, etc., can be removed by a magnet, attached to an automatic grader or to the nibbing machine.

It is not often necessary to wash the beans, though this treatment is employed by some manufacturers with poor and dirty samples. The same advantages, as would be attained by washing, would be gained by the treatment of the beans with antiseptic solutions of boracic acid, if the cacao is to be stored for any length of time, or with improving solutions of caustic potash or soda.\*

Many marks of beans, among which may be mentioned those of Haiti and Samoa, and some of the African cacaos, are distinctly earthy in taste. This unpleasant flavour is derived usually from the system of curing and is concentrated in the husk. Any quantity of mould on the cacao is apt to impart a similar flavour to the interior of the beans, and it is on such occasions that advantages may be derived by washing the beans in water or in a 5 per cent. boracic acid solution at about 35° C. The necessity of thoroughly drying the cacao, washed with water or aqueous solutions after roasting, should be emphasised, otherwise a further development of mould would leave the beans in a worse state than before treatment.

Another practice, not uncommon with manufacturers, is to submit the beans to a post-fermentation process, by soaking the beans in water for forty-eight hours and drying off at 45° C., an operation which, in the case of unfermented beans with gray and unattractive shells and with a violet

\* *Vide* Chapter XIII.

and bitter interior, is said to improve greatly the appearance of the bean and the colour and flavour of the kernel.

If the beans are to be stored for any length of time before roasting, it is advisable to make a careful examination of them on their arrival into the factory, as they may have become damp during transport, in which case the formation of mildew will rapidly bring about deterioration of their flavour, or they may have been infected with the eggs of the moth, so common in cacao storehouses, which develop into maggots in early spring, causing considerable damage to the beans.

If the beans are damp, they should be spread out on the open floor of a well-ventilated and warm room, though, if they are to be treated with water or aqueous solutions, as already mentioned, the drying may be postponed till after treatment.

Cacao that has become infested with moth or maggot should be picked over by hand, and the worm-eaten beans removed, whilst it is a good plan to expose the rest of the beans to the disinfecting rays of the sun, if possible.

All beans are liable to be attacked by the cacao moth, which lays its eggs inside the outer shell of the bean, and even the finest grades of cacao are not immune. The maggots, as they hatch out, feed upon the contents of the bean and, when sufficiently developed, crawl through the sacking and swarm over the ceiling and walls of the storehouse.

If the maggots are not destroyed, they develop in the summer months, after the usual pupa stage, into moths which, in their life cycle, deposit their eggs in the beans, if such are stored loose, or on the surface of the bags containing the cacao.

The moths may be got rid of by placing large shallow pans of water near and around a lamp in the storehouse, to

which they are attracted and, becoming stupified by the glare, fall into the water and are drowned.

In cases where the eggs have been deposited on the sacking, the maggots are less easy to destroy in time to prevent them from attacking the beans, as the newly-hatched larvæ seem to make their way through the sacking to the beans and, when more developed and after the damage has been done, crawl out and swarm over the room. At this stage, they can be attracted to the water in the pans, used for destroying the moths, and large quantities can be drowned in this way. Spraying the sacks with formalin solution is also very efficacious.

A systematic sorting of the beans and bags before storing is undoubtedly the best remedy against the depredations of moth and maggot, whilst good ventilation and frequent lime-washing of the walls of the storehouse will greatly aid in keeping these pests in check.

Further information regarding the cacao moth, its life-history and methods for destruction, can be found in the records of the Association of German Chocolate Manufacturers.\*

### Sorting.

In the most primitive form of sorting, and when only small quantities of cacao are to be handled, manual labour is employed, but, in large factories, it is obvious that such a process would be most costly.

Consequently, the makers of plant for the manufacture of cocoa and chocolate have turned their ingenuity to devising machinery by means of which a large bulk of beans can be sorted and cleaned. The principle underlying all sorting machines is the progress of the beans through a series of sieves of varying meshes, which are usually provided with a rocking motion, in order that full advantage of their surface may be taken.

\* G. Reinhardt, *Korr. Verb. deutsch. Schokoladefab.*, 1891, vii.

The top and first sieve is of a fine mesh which will allow only very small and shrivelled beans, small pieces of wood, stones and dust, to pass through. The beans, in falling to the next sieve, may be winnowed by a power-driven fan in



FIG. 1.—Cacao Bean Sorting and Cleaning Machine, with Sorting Band. (See p. 134.)

*By permission of Messrs. Joseph Baker, Sons, and Perkins, Ltd.*

such a way and direction that hollow beans, pieces of sacking, straw, etc., which are lighter than the smallest desirable bean, are blown into a receptacle leading to the passage where the first sievings are descending to the waste outlet.



According to the number of grades required, usually two or three, the beans must pass over a number of sieves of different meshes, each sieving being led to a separate outlet. Subsequently, the grades may be picked over by hand, to remove any defective beans or foreign material that may have escaped the sieving.

• It may occur that it is only necessary to pass the beans over the first mesh, if the sample is fairly even in size, in which case they are run from the sieve on to a travelling band, where they are rapidly freed from shrivelled beans and foreign refuse by operators standing in front of the beans as they are carried by.

The machine, illustrated (Fig. 1), made by Messrs. Joseph Baker, Sons, and Perkins, Ltd., is constructed so that the beans are fed from a hopper into an inclined revolving cylindrical sieve, divided into six divisions of different meshes. The rough beans, in passing over the first division, are rid of dirt, dust and small foreign matter, while the remaining divisions grade the beans into various sizes.

It is obvious that a certain amount of foreign matter, of similar size to the corresponding grades, will be found in the different receptacles among the beans, and, to obviate this difficulty, a band of canvas can be arranged to take the beans falling from one or two of the different divisions to be picked over by hand. Such a machine, occupying a floor space of  $12\frac{1}{4} \times 10\frac{1}{2}$  feet, is  $6\frac{1}{2}$  feet high and is fitted with a sorting band and capable of large output.

## CHAPTER XI

### ROASTING OF CACAO ANALYSES OF BEANS, NIBS AND SHELLS, BEFORE AND AFTER ROASTING

AFTER the process of sorting, the beans are next submitted to roasting, an operation of great delicacy and of far-reaching effects.

In the earliest days in the history of chocolate, the Mexicans used to roast their cacao in earthenware vessels over an open fire, and, as is the case with the Chinese cooks of the Philippine Islands of the present day, they succeeded in reducing their appliances for the manufacture of chocolate to a portable form.

A vessel, for roasting the beans, and a pestle and mortar, for pounding the roasted nibs, comprise all the apparatus necessary for the preparation of chocolate, and, in spite of the great advances made in the machinery for the manufacture of cocoa preparations on the large scale, the principles underlying the modern machines are the same as those of the primitive apparatus employed by the Chinese cook.

The advantages to be derived by roasting are :—

(1) Development of the aroma of the beans, brought about by changes occurring in the volatile oils at a high temperature.

(2) Modification of the colouring matters of the bean.

(3) Gelatinisation of, or rendering soluble, some of the starch granules.

(4) Oxidation of the tannin and other astringent matters.

(5) Drying of the husk and bean, so that the former can be readily removed, and the latter be freed from moisture,

the presence of which would hinder the complete mixing of the fatty nibs, when crushed, and tend to destroy the keeping qualities of the cocoa and chocolate, prepared from the damp nibs.

The following table shows the changes taking place in the composition of the bean during roasting, as given by two observers :—

TABLE XXI.—*Analyses of Nibs of Cacao Beans, Before and After Roasting.*

	Arriba		Guayaquil Machala		Caracas		Trinidad		Trinidad.		Caracas	
	Before	After	Before	After	Before	After	Before	After	Before	After	Before	After
Moisture	8.35	8.52	6.33	6.25	6.50	7.48	6.20	7.85	6.34	2.63	6.63	5.60
Fat	50.39	50.07	52.68	52.09	50.31	49.24	51.57	48.14	43.66	41.89	36.81	37.63
Albumen	19.40	16.84	12.05	15.58	17.23	19.62	15.80	10.22	11.90	12.02	10.59	12.36
Theobromine	0.35	0.30	0.33	0.31	0.77	0.50	0.40	0.42	0.85	0.93	1.13	0.99
Starch	5.78	9.10	8.29	11.50	7.65	9.85	11.07	8.72	4.98	5.70	3.81	6.07
Cellulose or woody fibre	2.70	2.59	2.40	2.59	2.61	2.54	2.63	3.84	13.01	10.64	16.35	11.69
Ash	4.12	3.89	4.11	3.75	4.17	3.92	2.87	4.12	3.60	3.70	4.30	5.03

The first four of these analyses are by Zipperer, made in 1886; the last two are selected from Ridenour's work on cacao and chocolate in the *American Journal of Pharmacy* in 1895.

It is somewhat difficult to reconcile the results obtained by these two investigators, for, not only are the values obtained for albumen, starch, cellulose and theobromine widely different, but there are shown sometimes an increase in moisture and a decrease of fat after roasting.

It is possible, however, that some of the proteid matter or albumen, as estimated by Ridenour, may be included in his values for extractives, and it is noticeable, also, that the sum of that observer's figures for starch, cane-sugar and glucose more readily approaches to Zipperer's figures for starch. Cane-sugar, glucose and extractives have not been included in the above table, though Ridenour estimated

TABLE XXII.—Analyses of Cacao Beans, Before and After Roasting. (Weigmann.)

	Caracas		Puerto Cabello		Iruand		Arriba		Machala		Port au Prince		Surinam								
	I.	II	I.	II.	I.	II	I.	II	I.	II	I.	II	I.	II							
Moisture	7.77	6.73	5.03	8.08	5.92	4.03	7.47	6.32	4.49	8.25	8.17	4.16	4.17	7.07	3.40	7.77	7.14	3.10	7.53	5.26	3.92
Fat.	45.54	45.65	50.37	46.61	46.55	58.83	44.62	45.06	54.17	45.15	45.53	49.86	45.93	46.21	54.64	48.35	48.35	55.31	44.74	45.53	55.81
Total nitrogenous matters	14.13	18.66	14.05	13.50	13.65	13.25	14.06	13.68	12.90	15.37	15.73	15.81	14.06	15.76	13.62	14.56	14.11	14.19	13.65	14.25	13.37
Theobromine	1.48	1.92	1.57	1.51	1.52	1.65	1.31	1.44	1.25	—	—	—	—	1.40	—	—	—	1.75	1.66	1.75	1.74
Carbohydrate	19.40	22.75	21.76	22.92	24.75	24.53	25.39	26.40	21.38	22.73	22.45	22.81	23.19	24.17	21.55	21.50	21.64	19.82	26.16	26.60	29.07
Starch																					
Cellulose	6.10	5.40	3.60	4.43	4.74	3.60	4.55	4.92	3.51	4.38	4.17	3.48	4.36	4.01	3.11	5.19	4.35	3.57	4.30	4.80	2.92
Ash	4.91	4.49	3.87	4.28	4.28	3.88	3.48	3.55	2.88	3.80	3.43	3.74	4.09	3.85	3.49	4.15	3.80	3.41	3.16	3.33	2.93
Silica	2.06	1.92	0.32	0.18	0.05	0.06	0.10	0.07	0.07	0.14	0.10	0.14	0.22	0.23	0.13	1.45	0.41	0.34	0.13	0.14	0.08
Total nitrogen	2.26	2.17	2.25	2.16	2.19	2.18	2.25	2.19	2.16	2.40	2.32	2.23	2.25	2.17	2.15	2.33	2.20	2.27	2.19	2.29	2.14

their content in the bean, and they may be found in the *American Journal of Pharmacy*, vol. lxvii., p. 202.

The important changes that occur, during roasting, are made apparent in the analyses of beans, before and after roasting, given in the preceding table, which shows in column I. the whole raw bean; II. the whole roasted bean, and III. roasted "nibs," reduced to paste.

The form which analysis of nibs usually takes in modern work is illustrated in the next table,\* in which it will be noticed that the alkalinity of the ash and percentage of fibre figure prominently :—

TABLE XXIII.—*Analyses of Nibs of Roasted Cacao.*

	African	Grenada	Guayanquil	Trinidad.	Caracas	Bahia	Ceylon
Mineral matter, total	2.52	2.60	3.16	2.73	3.24	2.68	3.81
Mineral matter, soluble	0.98	1.04	1.32	0.95	1.58	1.22	1.66
Silicious matter	0.05	0.03	0.04	0.08	0.08	0.05	0.03
Alkalinity of mineral matter as K <sub>2</sub> O	0.38	0.55	0.53	0.44	0.74	0.51	0.67
Cold water extract	11.80	9.80	11.40	12.00	—	9.50	11.90
Nitrogen	1.84	2.26	—	2.32	—	1.98	2.44
Fat	50.20	50.80	—	55.70	—	44.40	50.20
Fibre	—	2.94	—	2.48	—	—	2.36

The reason for the inclusion of figures showing the alkalinity of the ash and the percentage of fibre is to detect addition of materials which should be foreign to pure cocoa and chocolate, the former providing information as to whether the cacao has been treated with alkali for making the so-called "soluble" cocoa, described more fully in a later chapter, the latter to detect the percentage of husk which has been allowed to remain in the roasted nibs, and which should be kept at the lowest possible figure in the manufacture of good cocoa and chocolate.

\* N. P. Booth, *The Analyst*, 1900, xxxiv., 143.

Analyses of the shells, by the same author, show the following results :—

TABLE XXIV.—*Analyses of Shells of Roasted Cacao.*

	African	Guayaquil	Ceylon
Mineral matter, total	5.63	8.19	6.61
Mineral matter, soluble	3.53	5.25	4.78
Silicious matter	1.79	1.35	1.00
Alkalinity of mineral matter as $K_2O$	2.63	3.36	2.54
Cold water extract	20.40	24.60	20.70
Nitrogen	2.91	2.13	2.40
Fat	3.50	5.90	3.10
Fibre	12.80	12.85	12.80

Boussingault, in his book, "Le Cacao et le Chocolat," gives, among others, analyses of cacaos before and after roasting, which will be found in Table XXV.

From these results, unlike those shown in Table XXI., it would appear that a very considerable increase in the

TABLE XXV.—*Analyses of Nibs, without Shell and Germ, Before and After Roasting.*

	Puerto Cabello		Martinique		Guayra	
	Before	After	Before	After	Before	After
Water	7.00	5.00	7.50	2.00	7.00	4.00
Fat	40.36	45.23	41.20	45.56	45.96	49.26
Ash	3.75	3.05	2.75	2.90	4.00	3.70
Nitrogen	2.18	2.19	2.25	2.32	2.18	2.20
Albumen	13.60	13.70	14.50	18.00	13.60	14.40

percentage of fat occurs on roasting, a fact which cannot entirely be accounted for by the loss of water which takes place at the same time. These figures, however, bear out experiments, frequently made by the author, who has recorded increases in fat-content, over that of the raw beans, during roasting to as high a temperature as  $145^{\circ}C.$  for as

long as thirty minutes. The temperature of the roast drops, of course, on admission of the beans (to about  $110^{\circ}\text{C}.$ ), and the long roasting results in burnt cacao. There is a marked increase in fat up to fifteen minutes' roasting, and, after that time, the fat begins to decrease, probably due to the absorption by the husks and to subsequent destruction by burning.

The percentage of theobromine is also affected by the temperature of roasting, thus :—

Before roasting, certain nibs yielded 1.11 per cent. theobromine.

After roasting at  $120^{\circ}\text{C}.$  for fifteen minutes, the same nibs yielded 0.77 per cent. theobromine.

After roasting at  $230^{\circ}\text{C}.$  for eight minutes, the same nibs yielded 0.25 per cent. theobromine.

#### **Method of Roasting.**

The roasting of cacao is conducted in an apparatus and manner similar to those employed in roasting coffee, though the temperature is not allowed to rise so high as in the latter case.

It is assumed that the beans have been graded and sorted, the varieties separately, if the beans are of different sizes, or together, if they are more uniform. If the varieties of cacao differ greatly in the size of the beans, they will be roasted separately; if similar, then a combined roast may be made, though an experimental roast is considered advisable.

The object of roasting is to facilitate the removal of the husk and to develop aroma. A temperature about  $100^{\circ}\text{C}.$  may be employed, if a partial roast is to be made, as, at this temperature, the husks are rendered just sufficiently friable to be removed readily. A fully roasted bean may require  $135^{\circ}\text{C}.$ , in order to develop its aroma to the best advantage, but, in any case, the process must be continued until the

beans have lost their vinegar-like smell which is most difficult to remove at later stages.

Roasting may be carried out partially or completely, according to the result required. It should be remembered that, when fully roasting, it is easier to rectify an under-roasted than an over-roasted bean, as the burnt flavour of the latter is not only unpleasant but very persistent. Experience alone can dictate whether the beans have been correctly roasted for any particular chocolate required. Good mechanical roasters are, however, as essential to success as a good supervisor, seeing that, at the temperature employed (for full roasting  $135^{\circ}\text{C}$ . is not often exceeded), it requires very little excess of heat to spoil the charge of beans.

Manufacturers of roasters are often not aware of the narrow margin lying between properly roasted and over-roasted beans, with the result that direct access of fire-gases is often allowed to the beans, and facilities for quickly discharging the roast are not provided. It is, of course, necessary to remove the steam and fumes from the interior of the roasting cylinder, but, in the best roasters, an exhaust apparatus is fitted for the purpose. Externally-heated roasters are the only kind to be recommended, and those so heated by gas are more under control than those heated by open fire. Easy access to the interior of the roasting drum, for purposes of thorough cleaning, is most desirable, as shells of previously roasted beans are sure to adhere to the side and, becoming burnt in subsequent roasts, will affect adversely the aroma of the resulting cacao. Especially is this the case with beans treated with alkaline solutions.

After roasting to the required degree, it is necessary to cool down the beans as quickly as possible, in order to prevent further roasting by their self-contained heat. Exhaust coolers are, with great advantage, fitted to the roasters



and usually comprise a large trough or box, lined with sheet steel, with a perforated false bottom on to which the beans are discharged from the roaster. Cold air is then drawn through the mass of hot beans, by means of a fan, to the outside of the building, and the whole is rapidly brought down to a moderate temperature.

#### Roasting Machines.

In France, the most favoured roaster is the "Sirocco," which consists, essentially, of one spherical receptacle revolving in another. Between the two spheres pass the hot flue gases from a coke stove, set apart from the roaster itself. The hot gases are circulated by means of a forced draught. The beans are introduced into a hopper at the top of the roaster, and, when the inner receptacle is in position, the charge is let down into it, the flues opened, and the sphere revolved.

In order to ascertain the progress of the roasting, a sampler is fitted into the axle which, being hollow at this end and open on one side to the inner chamber, enables beans to be withdrawn at various stages in the operation.

Beneath the roaster is fitted a large circular open metal trough in which can be made to revolve a series of fans or brushes that quickly cool the beans let down into it, after the roasting is complete. The bottom of the trough is usually perforated and attached to an exhaust fan which draws cool air through the hot beans.

Other systems of dry roasting are based on the same principles, though, frequently, the flue gases are allowed access to the beans direct, a course which, whilst capable of giving satisfactory results, can only be recommended where constant supervision is given during the operation, as the beans roast more quickly and are, consequently, in greater danger of being over-roasted. Where such a system is in use, the greatest care should be taken that the coke fire,

supplying the hot gases, is quite bright and clear, and free from any smoke which might taint the beans.

A machine, made by Messrs. Lehmann, of Dresden (Fig. 2), has been designed for giving very quick roasts and consists of a drum revolving over an open fire, while the process is materially assisted by the passage through the

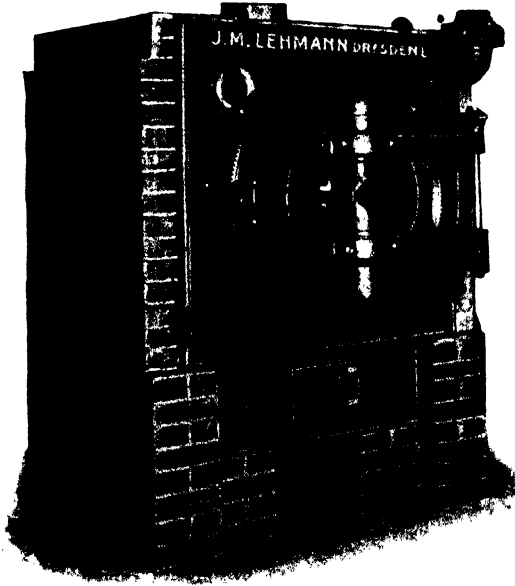


FIG. 2.—Cacao Bean Roaster for Open Fire. (See p. 143.)

*By permission of Messrs. Bramish & Co., London.*

drum of a hot air current, entirely unconnected with the fire gases. By reason of the hot air displacing the vapours coming off the beans and forcing them through the ventilation pipe in front of the drum, the roasting is considerably accelerated.

This machine is charged through a hopper at the back

and is made to discharge automatically, whilst the drum is designed to revolve while charging, an advantage which

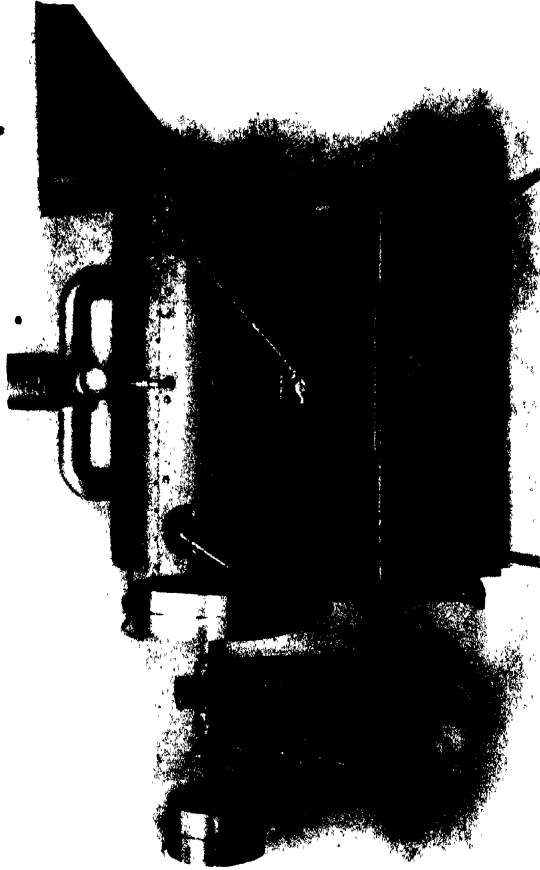


FIG. 3.—Improved Gas Cacao Roasting Machine. Externally heated, either by town or producer gas. (See p. 145.)  
By permission of Messrs Joseph Faler, Sons, & Perkins, Ltd.

prevents the over-roasting of the beans that first come in contact with the hot metal.

The sampler is placed in a similar position to that of the "Sirocco" roaster, namely, in the drum axle.

An ingenious system of rapid roasting of ordinary and medium grades of hard-shelled beans is one embodied in a roaster made by Messrs. Joseph Baker, Sons, and Perkins, Ltd. In this design, the beans are fed through the end of a cylinder and continually fall, until roasted, through hot gases, supplied by burning an admixture of town or "producer" gas and sufficient air to ensure complete combustion. Movable coolers, with perforated bottoms, can be supplied with this roaster, and, after receiving their charge, they can be wheeled away to an exhaust fan for cooling. The whole machine is simple, strong and capable of a large output, as the process of roasting is much shorter than in the externally-heated machines which are next described.

Like the "Sirocco," the externally-heated roasters consist of one drum or cylinder revolving in another. The heat is supplied to the outside of the inner drum or cylinder by a series of gas burners, and an exhaust pipe carries off the steam and fumes from the roasting beans.

Exhaust coolers can be fitted to these machines, so that, when the cacao is ready to be discharged, the cooler is run underneath, and the beans are allowed to fall into it. It is then run out on rails to the fan which draws cold air through the hot beans into the space beneath the false and perforated bottom, with which the coolers are provided, and thence, through the fan, to the outside of the building.

Such a machine, adapted for town or "producer" gas heating, fitted with a cooler, and of a capacity of 200 lbs. *per* roast, would occupy a floor space of  $8\frac{1}{2}$  feet by  $5\frac{1}{2}$  feet (Fig. 3).

The roasting process is the most important in the course of manufacture of cocoa and chocolate, and special care should be bestowed upon it, as no correction can afterwards be made if a bad roast, especially an over-roast, has been produced.

## CHAPTER XII

### NIBBING, HUSKING, AND WINNOWERING THE ROASTED • CACAO ANALYSES OF NIBS AND SHELLS, AND THEIR PROPORTIONS IN THE ROASTED BEAN

THE cacao beans, after having been roasted and rapidly cooled down on one of the exhaust coolers already described, are next taken to be husked or deprived of their shell. This process briefly consists of cracking the beans between two set rollers and, subsequently, sieving and winnowing. In practice, the broken pieces are made to pass over a series of sieves of different mesh, while an air draught, travelling in an opposite direction to the nibs, blows the husk from them.

The beans should not be allowed to get stone-cold before passing through the rollers, as the cold, crisp kernel is liable to be broken up into very minute pieces which, being of the same weight as the shell, may be blown away into the husk receptacle and cause considerable loss.

The quality of cocoa and chocolate is largely dependent upon the completeness of this process, for it must be realised that the nibs of cacao beans, *alone*, should be used for cocoa preparations and that the inclusion of husk must be looked upon as adulteration.

With modern machinery, there can be no excuse for the presence of any quantity of husk in a cocoa or chocolate, as, by a careful adjustment of the fans, an almost complete separation of nib and husk can be effected.

The problem of estimating the percentage of husk in cacao preparations has always been of great consequence, and Filsinger, one of the earliest investigators of cacao and

its preparations, describes a method of calculating the shell-content by a sedimentation method in glycerine and water. This process, however, is incapable of giving reasonably consistent results.\*

The determination of the quantity of cellulose, present in a cacao preparation, is some indication of the extent of cleaning, or adulteration, to which the beans have been subjected, as the greater the proportion of husk the higher will be the value for cellulose.

TABLE XXVI.—*Estimations of Cellulose in Cleaned Roasted Nibs.*

	Cellulose	
	For 100 parts cacao	For 100 parts cacao insoluble in water.
Sanchez	4.90	14.75
Bahia	4.76	14.64
Haiti	4.86	14.60
St. Lucia	4.50	13.80
Guadeloupe	5.03	15.15
Trinidad	5.00	15.27
Grenada	4.93	15.05
Maragnan	4.66	14.51
Carupano	5.11	15.61
Porto Plata	4.63	14.14
Caracas (small)	5.53	16.60
Mean	4.90	14.91

The above table shows recent results for the estimation of the percentage of cellulose, present in pure cacao paste or ground cleaned nibs, given by Dr. Bordas, Director of the Laboratories of the Ministry of Finance in France, at the Seventh International Congress of Applied Chemistry held in London, in 1909.

The estimation of cellulose alone, however, may be misleading, as extraordinary fluctuations occur among different varieties, as also among beans of the same origin. More precise and accurate figures may be obtained by taking

\* See also Chapters XXX. and XXXII.

into consideration also the quantity of ash, its alkalinity and its composition, and, especially, of the amount of silicic anhydride of which the husk contains a much larger quantity than the kernel.

The composition of the ash, obtained from various cacaos, is given by Bordas as follows :—

TABLE XXVII.—*Composition of Ash.*

	Ash		SiO <sub>2</sub> and sand.	Cl.	SO <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub> Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	P <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	CO <sub>2</sub> by difference.
	Sol.	Insol.										
Bahia	33.33	66.66	1.60	0.20	2.57	0.60	3.60	15.65	32.60	37.96	1.50	3.72
Haiti	30.26	69.74	7.34	0.48	2.66	1.93	5.18	14.15	27.65	30.41	2.63	7.57
St. Lucia	31.25	68.75	0.93	0.30	3.06	0.99	5.25	15.21	33.78	38.50	1.50	5.54
Guadeloupe	33.14	66.86	4.18	0.50	3.06	1.33	5.00	14.14	29.22	34.94	1.27	6.56
Tinidad	32.55	67.45	7.00	0.40	3.80	0.74	3.54	15.43	28.38	32.28	1.90	5.53
Grenada	31.03	68.97	1.50	0.35	3.51	0.25	3.82	17.62	32.94	33.06	0.26	5.70
Maragnan	27.36	72.64	0.60	0.30	4.00	0.58	4.46	17.74	31.28	38.00	0.16	2.88
Carupano	33.76	66.24	3.50	0.20	3.35	0.36	3.66	16.06	30.10	32.91	0.60	9.16
Porto Plata	32.10	67.90	0.53	0.25	3.10	0.65	3.40	16.00	33.50	38.30	1.50	2.77
Caracas (small)	34.12	65.88	7.00	0.20	3.10	1.82	4.30	13.00	25.60	32.50	2.50	7.08
Germ.	56.67	43.33	1.00	0.50	4.24	0.40	4.30	9.73	18.50	54.50	0.50	4.93
Shell (roasted)	43.15	56.85	19.10	0.80	2.50	3.60	4.72	7.40	8.50	4.50	3.50	0.38
Dust and small cacao	35.56	64.44	26.52	—	—	—	—	—	—	—	—	—

A very accurate method for detection of added husk to cacao by estimation of the pentosans, fully described in a later chapter, has been devised by Adan.\* The Committee of the Bromatology Section, at the 1909 International Congress of Applied Chemistry, suggested that the following resolution should be submitted :—

“The Congress, believing that the determination of pentose and pentosans affords an excellent criterion of the purity of cocoa powder, recommends the adoption of this determination in the routine analysis of cocoa.”

Adan's results are embodied in Table XXVIII.

From these figures, it will be seen that the inclusion of husk in cacao preparations, whether deliberate or accidental, should be readily detected by the chemist. The determination of the quantity present is less easy and has been discussed at length in Chapter XXXII.

\* R. Adan, *Internat. Cong. App. Chem.*, 1909, viii., c. 194.

TABLE XXVIII.—Analyses of Shelled Cocoa Nibs.

	Moisture.	Fat	Starch	Ash	Cellulose	Pentosans.			
						Grains or nibs		Shells.	
						Initial substance	Powder with 30 per cent fat	Dry substance	Substance with 10 per cent. moisture.
Arriba	8.27	45.15	5.83	3.88	4.48	1.71	2.79	—	—
Roasted Shells	8.52	50.07	9.10	3.89	3.70	1.29	2.19	—	—
Port au Prince	7.77	46.35	5.97	4.15	5.19	1.59	2.60	9.97	9.11
Roasted Shells	4.73	51.87	8.40	3.49	4.31	1.27	1.99	—	—
San Thomé	8.08	46.61	5.69	4.28	4.43	1.43	2.34	7.57	6.92
Roasted Shells	5.71	50.20	13.27	3.89	4.33	1.45	2.57	—	—
Caracas	7.77	45.54	5.48	4.91	6.18	1.56	2.71	8.49	7.92
Roasted Shells	7.48	49.24	9.85	3.92	4.24	1.19	1.86	—	—
Bahia	5.96	42.10	7.53	3.63	7.86	2.10	3.14	7.78	7.11
Roasted Shells	3.71	50.19	9.61	3.24	3.93	1.77	2.73	—	—
Soconusco	2.95	43.38	8.33	3.21	3.34	1.59	2.60	9.45	8.70
Roasted Shells	5.00	50.22	9.58	3.76	3.78	1.21	2.06	—	—
AVERAGE	6.43	44.44	8.22	4.00	4.78	1.53	2.47	9.96	8.21

The Association of German Chocolate Manufacturers offered a prize some years ago for a machine that would separate the nibs completely from the husk without allowing any quantity of the former to find their way into the waste receptacle. The problem was not so easy of solution as would be imagined, for the bean, when passing through the crushing rollers, is certain to break up into a number of different-sized particles, the smaller of which, being light, could not help passing along with the husk or being sieved out with the dust and finer particles of the shell.

As the outcome of this offer, many machines, claiming to do the required work, were quickly put on the market, and, though a large number failed when put to the test, several were taken up by the larger manufacturers.



**Method of Nibbing, Husking' and Winnowing.**

In all machines employed for nibbing, husking and winnowing, the first step is to crack the beans, freeing the nibs from the shell for subsequent separation. This is brought about by passing the cacao through two rollers, which are so adjusted to the size of the roasted beans that they do not crush too severely, but crack the outer crisp shell and enable the nibs, or portions of the kernel into which it is divided, to fall away.

The fragments pass into an inclined revolving cylindrical screen, divided into meshes of various sizes; the first division removes the dust and very small particles of cacao, while the remaining divisions grade the nibs according to their size. A current of air, capable of being regulated so as to effect the separation of husk from particles of the nibs, which may be of the same size as, but specifically lighter than, the shell, is supplied to each division by means of a separate fan.

The cylindrical sieve may contain any number of meshes of different sizes, according to the number and dimensions of the grades required. In the larger machines, there may be six or eight divisions, though the more usual number is three or four.

**Nibbing, Husking, and Winnowing Machines.**

In one machine, made by Messrs. Joseph Baker, Sons, and Perkins, Ltd., the beans are passed through two rollers, capable of being set to any required dimension to suit the size of the sample, or for the purpose of regulating the grade of the nibs. The crushed beans are carried to the top of the machine by an elevator and are discharged into an inclined cylindrical sieve. Separation of the husk from the nibs is efficiently carried out by fans, with independent air inlets, which can be separately controlled. The machine is made with six or eight divisions and the cylinder has a

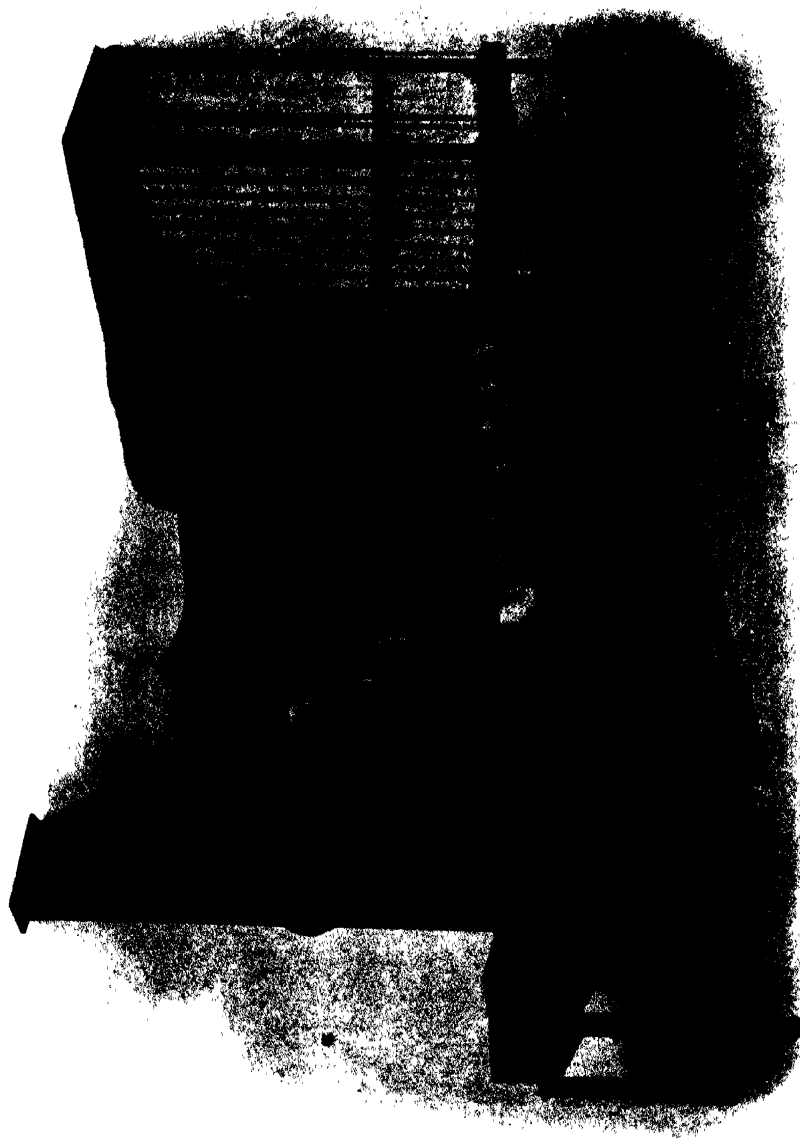
corresponding number of different meshes, whilst two of the sections are fitted with narrow longitudinal openings through which the germs pass, excluding the greater part of the cacao which passes over into later divisions.

Complete separation of the germ from the finer cacao is carried out in the germ separator. This separator is described later as an independent machine.

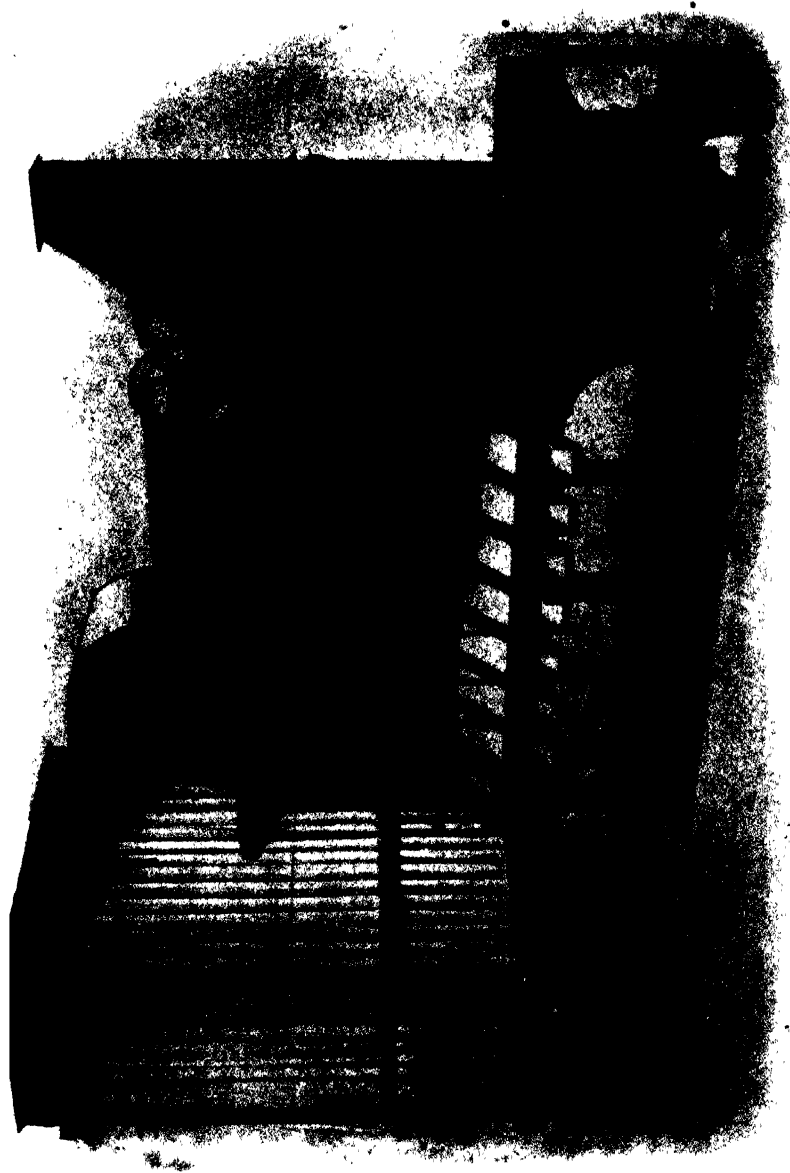
The heavier pieces of shell, to which some cacao has remained adhering, and the pieces which have not passed through any division of the sieve, fall, at the end of the machine, into a worm conveyer and are returned to be passed again through the rollers.

In the ordinary winnowing machines, the germ, the dead embryo or radicle of the seed, is not separated from the nibs at this stage, but, being of a hard, gritty and fibrous nature, and of coarse flavour, it must be removed before converting the nibs into cocoa powder or chocolate.

The machine, illustrated (Figs. 4 and 5), is the most up-to-date production of the firm of Messrs. Joseph Baker, Sons, and Perkins, Ltd. The front view shows, on the left-hand side, the hopper into which the beans are fed and the nibbing apparatus just below it. The cracked beans are elevated from the nibber up the upright tower on the left and pass down the chute on to a flat sieve which is kept oscillating. The nibs and shells both fall through the perforations of the sieve into collecting spouts, placed just beneath, which bring the nibs, etc., to the side of the machine over sheet-iron baffle plates. By means of the fan, situated at the further end, the shells are drawn upwards and delivered to the spout to be seen on the extreme left of the back-view of the machine. The fine dust, etc., drawn up also by the fan, is blown over into the settling chamber, which can be seen most clearly on the left of the illustration of the back-view. The settling chamber consists of a number of calico tubes which allow the air, but not the dust, to escape, the latter



**FIG. 4**—New and improved Nibbling, Husking and Winnowing Machine. Front view. (See p 151 )  
*By permission of Messrs. Joseph Baker, Sons and Perkins Ltd.*



**FIG. 8.**—New and improved Nibbling, Husking and Winnowing Machine. Back view. (See p. 151.)

*By permission of Messrs. Joseph Baker, Sons and Partners, Ltd.*

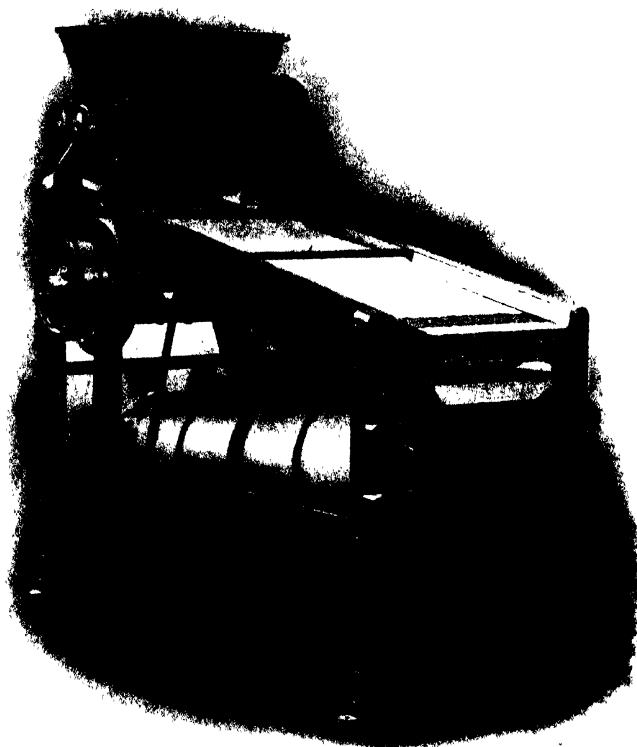


FIG. 6 - Cacao Germ-separating Machine. (See p. 155.)

*By permission of Messrs. Joseph Baker, Sons, and Perkins, Ltd.*

falling into a chamber at the bottom, whence it is delivered to the end chute by a worm conveyer.

#### **Germ-Separating Machine.**

The separation of germ is usually effected in a cylinder, either perforated with longitudinal slits through which the

germ and small cacao pass, to be separated again by a system of winnowing, or the cylinder may be indented in such a manner that the cavities are capable of taking the small fragments of cacao, but will not hold the elongated germ, or *vice versa*.

The cacao grains in the cavities pass upward beyond a light scraper, until they reach a certain elevation, when a brush removes them from their holes, and they fall into a trough, whence a worm conveyer carries them away. The germ, on the other hand, not fitting into the cavities, remains below the scraper and, by the tilt of the cylinder, is made to pass in an opposite direction to the cacao, complete separation being thus effected.

The nibbing, husking and winnowing machine, already described, fitted with a germ separator, will occupy approximately 12½ feet by 7½ feet floor space, and be about 9 feet high.

The following figures, adapted from those given by Hart,\* as the result of some experiments on the loss, etc., during manufacture of cocoa powder, are interesting. Only small samples were dealt with, however, and the percentage loss is, consequently, higher than it would be on the manufacturing scale.

TABLE XXIX.—*Losses in Preliminary Preparation of the Beans for Manufacture.*

	Fine clayed Venezuela	Fine Trinidad	Ordinary Trinidad
Before roasting	100	100	100
Weight of husk	16.8	16.3	15.3
Loss (apart from husk)	7.7	5.1	6.7
Weight of dry cacao after removal of fat	55.0	56.0	54.8
Weight of fat extracted	12.3	16.4	16.1
Loss during grinding and expression of fat	8.2	6.2	7.1
Total loss on manufacture (including husk)	32.7	27.6	29.1

\* J. H. Hart, "Cacao," 1900, 113, and 1911, 255.

The figures have been calculated on 100 parts of bean and have been adjusted to suit the requirements of manufacturers.

Filsinger,\* from a sample of 100 parts of Machala cacao, containing an equal proportion of large and small beans, obtained the following divisions: 70 parts large nibs, 9.2 parts medium nibs, 0.8 parts radicles or germ, 10 parts husk, 4 parts cacao waste, 6 parts loss. The four parts of cacao waste, on further sifting, yielded about 30 per cent. each of nib, husk and cacao dust, the remainder being waste and loss.

The great improvements which are said to have taken place in recent years, both in the quality and yield of the beans, and in the machinery for preparing them for chocolate manufacture, are not very apparent from the following figures, showing the losses sustained during preparation, given by Bernhardt,† in 1889.

TABLE XXX. - *Losses in Preliminary Preparation of the Beans for Manufacture.*

	Mean	Maximum.	Minimum.
Sifting . . .	2.80 per cent.	5.49 per cent.	1.10 per cent.
Picking . . .	0.80 ..	2.09 ..	0.25 ..
Roasting . . .	5.51 ..	7.05 ..	4.61 ..
Cleaning . . .	13.00 ..	16.04 ..	10.08 ..
Total loss . .	22.11 ..	25.78 ..	16.76 ..

The next table shows results obtained in the author's laboratory at Messrs. Peek, Frean and Company, Ltd., London, from roasting and hand-picking cacao beans. The proportions of shell, nib and germ, and the loss on roasting, found in a few varieties of cacao from a large number of samples, are given. For valuation of cacao beans for the

\* Filsinger, *Zeitsch. öfent. Chem.*, 1898, 810.

† Bernhardt, *Chem. Zeit.*, 1889, 32.

manufacture of different cocoa preparations, a similar examination is recommended, and, provided the cacao beans have been fairly sampled, very valuable information for the factory can be obtained by these means.

TABLE XXXI.—*Losses on Roasting Cacao Beans, with Percentages of Husk and Germ.*

Cacao.	Loss on roasting Per cent.	Husk. Per cent.	Germ. Per cent.
Trinidad . . . .	2.70-7.30	11.10-16.82	0.72-0.82
Grenada . . . .	3.58-7.00	11.86-15.38	0.60-0.90
St. Lucia . . . .	3.57-4.46	12.80-15.00	0.65-0.78
Puerto Cabello . .	1.70-5.00	12.95-15.55	0.75-0.91
Venezuela . . . .	4.50-7.25	15.50-20.30	0.81-0.92

Arpin\* has given the following table:—

TABLE XXXII.—*Proportion of Nibs, Husk and Germ in Roasted Cacao Beans.*

Cacao	Nibs. Per cent.	Husk Per cent.	Germ. Per cent.
Carupano . . . .	85.10	14.10	0.58
Chuao . . . . .	87.70	11.70	0.55
Ibarra . . . . .	86.00	13.40	0.53
Madagascar . .	91.20	8.40	0.48
Puerto Cabello . .	84.70	14.50	0.54
Trinidad . . . .	84.80	14.60	0.67
Mean . . . . .	86.60	12.80	0.56

In this connection, it is not without interest to record that Harrison has shown that, in fresh beans, the proportion of kernel to cuticle is about 85 : 15 in those of Calabacillo variety, and 91 : 9 in Forastero cacaos. It is presumed that Harrison examined high-grade Forastero cacao, since the figures, shown, for the cuticles are very low for average beans of that variety.

\* Arpin, *Ann. des Falsif.*, January, February, 1917, xcix., p. 10-14.



Other figures, obtained by different workers with the beans as they appear on the market, are given in the next table.

TABLE XXXIII.—*Husk in Different Varieties of Cacao Beans, Cleaned and Roasted.*

Cacao	Husk Per cent.	
	Cleaned beans.	Roasted beans.
Puerto Cabello . . . . .	15 00—17 75	14 30—15 50
Arriba, ordinary . . . . .	15 44	12 40—16 00
Arriba, superior . . . . .	13 70	12 00—14 50
Caracas . . . . .	12 40—16 90	12 80—15 56
Guayaquil . . . . .	13 00—24 00	10 30—12 00
Trinidad . . . . .	14 05	11 20—13 60
San Thomé . . . . .	11 30	10 00—11 30
Ceylon . . . . .	8 90	10 60—11 00
Bahia . . . . .	—	9 60
Cameroon . . . . .	8 00—13 20	—
Samaná . . . . .	12 10	—
Cuba . . . . .	14 70	—
Harti . . . . .	14 20	—
Machala . . . . .	13 80	—
Bahao . . . . .	14 00	—

It is possible to remove, by the use of good husking and nibbing machines, 10 per cent. coarse shells and some 2—3 per cent. of finer *débris*, whilst about 1—2 per cent. of the finest particles find their way into the nibs. It is not possible to remove every trace of the husk, though 95 per cent. of the total quantity is capable of elimination, if care is expended and the machines are efficient. A certain quantity of the inner fine membrane adheres very closely to the nibs and cannot be removed by winnowing or sieving. Cacao mass (the nibs after milling) will show up to 2 per cent. of husk, and cocoa powders, with 30 per cent. of fat, will often be found to contain as much as 3 per cent., though, in both cases, a higher figure is usually recorded.

At the Congress of Cocoa and Chocolate Makers held at Berne, in 1911, it was suggested that the provisional stan-

dard for cacao mass, cacao paste or bitter chocolate, should be as follows :—

“To consist of the products obtained by grinding of roasted and decorticated cacao beans deprived of their husks, and free as far as possible from germ and embryo. According to the use to which it is to be put, it is permitted to add or remove a variable proportion of cacao butter. Any added flavouring must be harmless.”

It was obvious from the discussion that the qualifications were included to meet the requirements of certain makers and vendors of bitter chocolate or cacao mass for certain purposes. The first paragraph should have stood alone. In addition to the first paragraph of the above, the following details might be included among the requirements of pure cacao mass :—

- (1) To contain not less than 45 per cent. of cacao butter.
- (2) To contain not more than 4 per cent. of ash or mineral matter, (ratio of soluble to insoluble ash not to exceed 2 : 3).
- (3) To contain not more than 6 per cent. of moisture.
- (4) To contain not more than 2.75 per cent. of pentosans.
- (5) To contain not more than 13 per cent. of starch. (which must be ascertained to be due to the cacao only, by means of the microscope).
- (6) To be free from added or included husk, cacao butter substitutes, starch, sugar, flavouring matters, etc.

The pentosan figure, shown, is merely an indication of the amount of fibre present and represents some 6 per cent. of crude fibre. This, by whatever method the estimation is made, is certainly a maximum figure for good bitter chocolate which, in the writer's opinion, should not contain more than 5 per cent. of crude fibre. The analyses of all good chocolates show that this figure (after allowance has been made for added sugar and cacao butter) is not exceeded in

the cacao mass from which the chocolate is made and is an indication, again, that the best chocolates are the result of care and attention to detail. Chocolate containing 10 per cent. of husk, or more, can readily be detected by a trained palate.

The other side of the question is efficiency and economy, and it is unnecessary to fine the winnowing and sieving down to such a point that any considerable quantity of small pieces of nibs pass away with the husk. The facts that foreign cocoa merchants used to pay high prices for the husks, especially from German factories, and that husks from certain factories in this country still command a high price show, among other things, that, often, a considerable quantity of valuable cacao, that might have been saved, still finds its way into the by-products of some chocolate factories. Delicacy and ease of adjustment of the machinery, used for this purpose, are, therefore, essential to an up-to-date chocolate plant, and it cannot be pretended that some firms, that the author knows, are as fully aware of the necessity and desirability of attention to this matter as its importance demands.

This is only one point, of many arising in the manufacture of chocolate, that belies the statement that "any fool can make good chocolate out of good materials," for, starting with the best cacao beans and the best of other ingredients, the finished chocolate can be ruined by allowing, through inattention or through imperfect machinery, too much husk to find its way into the nibs.

The need of removing the germ is as important as that of depriving the nibs of husk, since this is a hard body which, besides being valueless for flavour and slightly bitter, is extremely gritty when ground. The advantages, gained by installing a germ-separating machine, quickly repay the small expenditure. The author has made chocolate with a preponderating quantity of germ present and has found not

only a great deterioration in the natural aromatic flavour of the chocolate, but has experienced the greatest difficulty in refining down the grittiness due to the germ, a fact which may account for the occasional hard grit to be found in some of the higher quality chocolate, if insufficient attention has been paid to the removal of the germ. Quite recently, the author identified under the microscope several pieces of germ in one highly popular "fondant" chocolate in this country.

## CHAPTER XIII

### MILLING PREPARATION OF COCOA POWDER- EXPRESSION OF CACAO BUTTER "SOLUBLE" COCOA POWDERS

At this point comes the parting of the ways. If the beans have been fully roasted, the nibs, after passing through the nibbing, husking and winnowing machine, may go directly to the mills to be ground down to a paste for further treatment, to obtain—

(a) Cocoa powder.

(b) Chocolate.

If, however, the beans have been partially roasted (whether treated previously with alkali solution for making "soluble" cocoa powder or not), they have been dried only just sufficiently to facilitate the removal of the husk on the nibbing and husking machine. The nibs, in such cases, may be ground on granite rollers, since, being insufficiently roasted for passage through the mills, it is required to reduce them to a paste either for expression on the cocoa presses or for conversion into chocolate, which latter may also be accomplished by placing the nibs, with or without sugar, in a "mélangeur."

The methods of treatment of the nibs have now become so numerous that it is neither desirable nor necessary to discuss any particular process, but rather is it preferable to consider the special uses of every machine that has been found to be suitable for the general manufacture of cocoa and chocolate, individual processes being detailed as they arise.

The greatest advantages of milling are :—

- (1) Preliminary reduction of the nibs to a moderately fine condition.
- (2) Thorough blending of the mixed and blended nibs.
- (3) Convenience of storing block bitter chocolate, in preference to boxes of nibs.
- (4) Maturing of the cacao, when stored in blocks. •

In addition, there are numerous small advantages to be derived from milling, among others the saving of waste, if, by any chance, the blend has been incorrectly made or the beans badly roasted, materials which would have otherwise been incorporated with sugar and added cacao butter, if the nibs had been pounded down with the sugar in a “*melangeur*.”

Apart from this, milling prevents the living from hand to mouth in the factory, which would otherwise take place, and enables a stock of bitter chocolate, correctly blended, to be always ready to hand.

For the manufacture of cocoa powder, there can be no question of the great advantage to be derived from the reduction of the nibs to a moderately smooth homogeneous mass, in which most of the fat cells have been ruptured sufficiently to allow easy expression of the fat in the press.

### **The Cacao Mill.**

In modern mills, the nibs are fed into a hopper, so that they fall between a pair of revolving stones, the highest and first of a series through which the paste will have to pass during the grinding process.

As the nibs contain a large percentage of fat, they are readily reduced to the liquid state by the application of heat, and, to this end, the stones under which they pass are usually encased in steam-heated jackets. The greatest use of the steam-jacket is, however, at the commencement of milling, to heat up the cold stones, as, subsequently, the

heat of friction is sufficient to maintain the necessary temperature.

The grinding mill may consist of a single pair, double,

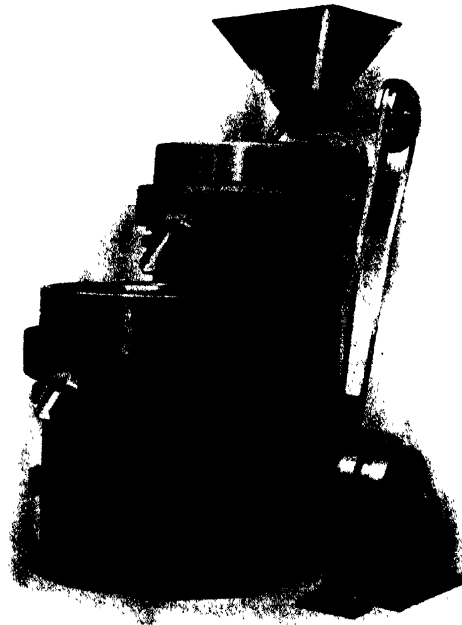


FIG. 7.—Double Cacao Grinding Mill, with 33-inch and 36-inch Top Runner Stones. (See p. 168.)

*By permission of Messrs. Joseph Baker, Sons, and Perkins, Ltd.*

or triple pairs of stones (Figs. 7, 8, and 9), the liquid cacao running from one to the other, in succession, as it undergoes reduction to a smooth consistency. The cacao mass flowing from the last pair of stones is in a semi-liquid state at a temperature of about 45° C.

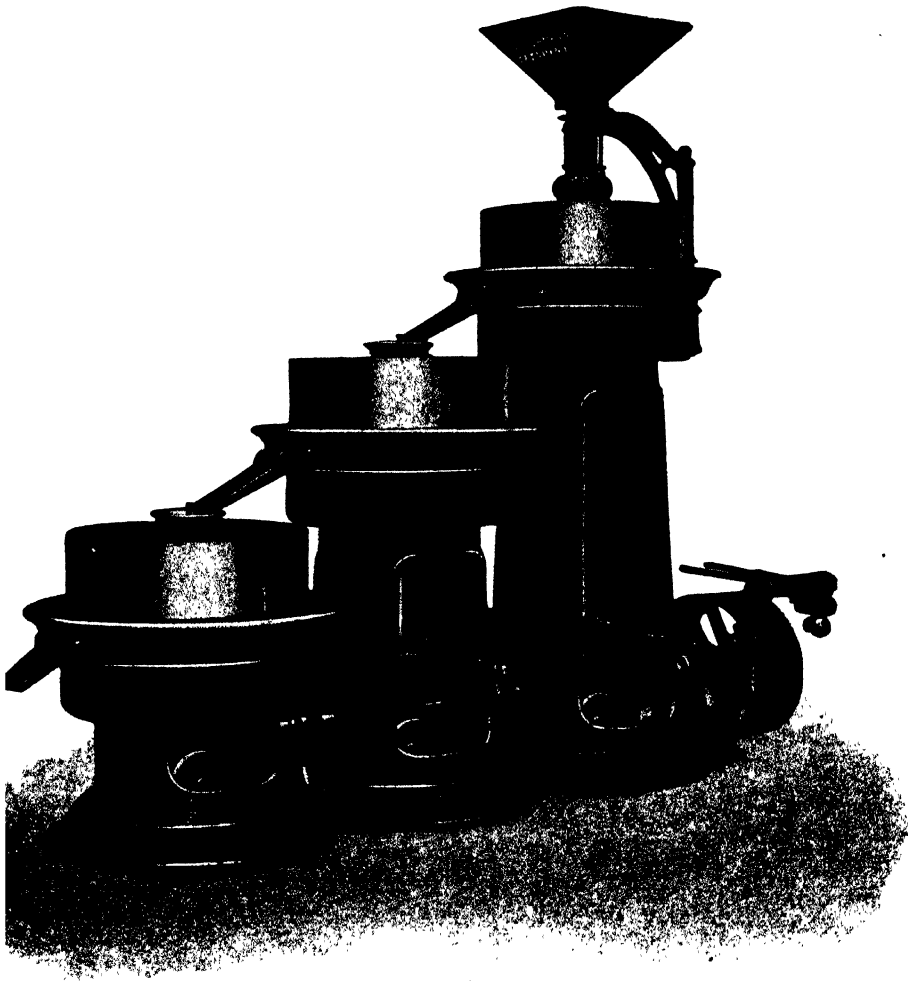


FIG. 8.—Triple Cacao Grinding Mill. (See p. 168.)

*By permission of Messrs. Bramigh & Co., London.*



The liquid that comes from the mills should be of such a consistency that the fat has been largely expressed from

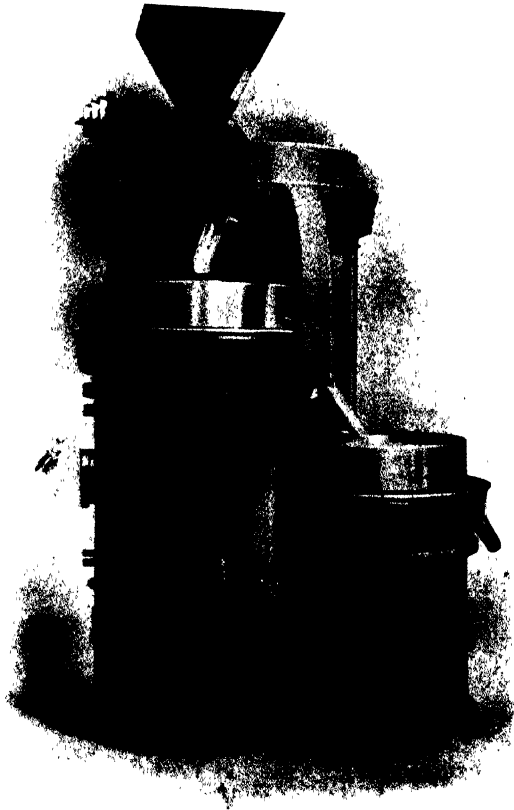


FIG. 9. —Triple Cocoa Grinding Mill, with 33-inch and 36-inch Top Runner Stones  
(See p. 168)

• By permission of Messrs. Joseph Baker, Sons, and Perkins, Ltd.

the cells, and the nibs reduced to the finest state of division without undue heating, during their passage through the stones. In this state, the success of the previous processes

can be well judged from the flavour. The cacao liquor is usually run into pans and cooled. When solid, the cakes may be removed and block-stacked, though it is doubtful if any great development or improvement of flavour occurs by keeping the bitter chocolate or cacao mass in this condition, at any rate for any length of time. It is certain, however, that the bitter chocolate, if kept in this way, should be stored in a cool, clean room, otherwise it will tend to melt and waste, and the cocoa moth is by no means averse to laying its eggs upon the blocks of chocolate, especially if they are undisturbed for some weeks.

An interesting point of some importance is to be noted here, especially by the chemist or the person who selects the sample for analysis. After leaving the mills and standing to cool in the pans, the liquid is inclined to separate into layers, the upper containing most fat, the lower part most of the heavier particles. It is not uncommon to find as much as 60 per cent. of fat in the upper layer, if the cooling to a solid state has been slow. For this reason, shallow pans are advisable and most commonly in use, since there might be a tendency, if larger and deeper pans were employed, to throw in more of the upper portions of the blocks for a mixing, with a consequent excess of fat. In sampling, this is a continual source of annoyance, and, when analysing or tasting the cacao mass in cake form, this point should not be overlooked.

Single, double, triple, or even five-stoned mills can be obtained from any maker of chocolate machinery, and, though their introduction is of comparatively recent origin, they are now looked upon as necessary additions to the cocoa and chocolate factory.

The reduction of the nibs to a fine homogeneous mass facilitates the incorporation of the sugar which is added to it in the "mélangeur," if chocolate is to be manufactured, and enables blends of different varieties of cacao beans,

separately roasted, to be made, so that a uniform and even mixture is obtained.

In the double cacao mill, illustrated, the capacity of output is about 1,000 lbs. of fine cacao liquor per ten-hour day. The machine occupies about 8 feet by  $3\frac{1}{2}$  feet floor space and is  $7\frac{1}{2}$  feet high.

• It has been proved that stones of too large a diameter often overheat the cacao and damage the flavour of the product. In the machine designed by Messrs. Lehmann, this danger has been obviated by very careful consideration given to the size limit, without curtailing the output. Only a slight warming of the stones, by means of the steam coils placed underneath, is required before starting. The feeding apparatus is adjustable and acts automatically, whilst the rate of progress of the nibs through the mill can be watched through a glass tube connecting the hopper to the first pair of stones and controlled at any time without difficulty.

In America, the triple mills are most popular, and the author saw several of Messrs. Lehmann's machines with stones between 40 and 44 inches diameter doing excellent work. The speed at which the fast pulleys are run is usually about 125 revolutions per minute, and the machine requires 15 horse-power.

A considerable amount of attention has been paid to the quality and size of the mill-stones used and to the speed with which they revolve, since unsuitable stones, or of too large a size or too quickly revolving, overheat the cacao with disastrous results. Here, too, the practical experience of years is to the hand of the manufacturer or the would-be cocoa and chocolate maker. The best stones are the French burrs, which, cut in a certain manner to ensure close grinding with maximum output and minimum heating, cannot be equalled when erected by the best engineers. Too much heating has been obviated, usually by allowing only the top

stones to revolve, but not a little of the success of the process depends upon the selection of the stones. The feed of the nibs to the stones must be nicely regulated, so as not to give too much work to the mills to do. Again, so much depends upon the work put into the machine that it is quite false economy to purchase mills because they happen to be cheap. It were better to exclude the milling process altogether than to work with machines that could not be relied upon or that were liable to spoil the cacao product.

Messrs. Joseph Baker, Sons, and Perkins, Ltd., have quite recently produced a "heavy weight" stone double or triple cacao mill which is known to give excellent results. The stones are of a finer grain than the French burrs and somewhat softer, but, owing to the introduction of special cutting plates and new methods of cutting the stones, a very fine cacao liquor is obtained. The objection to the necessity of re-dressing the stones rather more often than in the case of French burrs may be a serious one, but the smoothness of the resulting liquor is little short of remarkable, after experiences with some cacao mills on the market, and surely compensates for the extra trouble.

### **Preparation of Cocoa Powder and Expression of Fat.**

It has already been shown that cacao nibs contain some 50 per cent. of fat. This high content of fat makes the consumption of the beverage, prepared from pure cacao, too rich and indigestible for persons afflicted with weakness of the stomach, and, moreover, the presence of so large a quantity of fatty matter prevents the complete mixing of the cocoa and water used in its preparation.

In the early days of cocoa powders, the fat was extracted by submitting the nibs to a lengthy boiling in water, when the fat, rising to the surface, was skimmed off and purified, and the nibs, partially defatted, were placed in linen bags and subjected to pressure at a temperature about the boiling-

point of water, in order to remove further portions of fat and moisture. In this way, about 50 per cent. of the fat present was removed, but the resulting cocoa powder was of poor quality, having lost its fine aroma and colour from prolonged boiling in water, and was, moreover, liable to turn mouldy, owing to the presence of moisture which it still contained.

Other methods, still in existence, of lowering the percentage of fat in cocoa powder are by additions of starch and sugar. Such preparations cannot be termed "cocoa powder," which should consist of pure defatted cacao only, and which, when sold to the public, should bear on the packet or container printed notification of any added material.

#### **The Cacao Butter Press.**

The method now usually adopted in the preparation of cocoa powders is to run the cacao mass from the grinding mills direct into a press, where it undergoes extreme pressure, applied hydraulically, whereby some 60 to 70 per cent. of the fat is removed. Some manufacturers prefer to run the cacao liquor from the mills through a three- or five-roll refiner before passing it to the presses. In this way, the size of the cocoa powder particles is considerably reduced, thus increasing the yield of cacao butter and aiding the disintegration of the powder in the later process.

In the modern cup hydraulic press, of which an illustration is given (Fig. 10), the framework receivers and plunger dishes are of cast iron and are made with steam passages, so that the machine can be rapidly heated, ready for use. The receivers slide forward on rails for filling, and the capacity of a four-pot machine is about 90 lbs. per charge of four receivers. If a smaller charge is required to be expressed, any of the receivers may be fitted with iron dummies, so that one or all the pots may be temporarily put out of use.

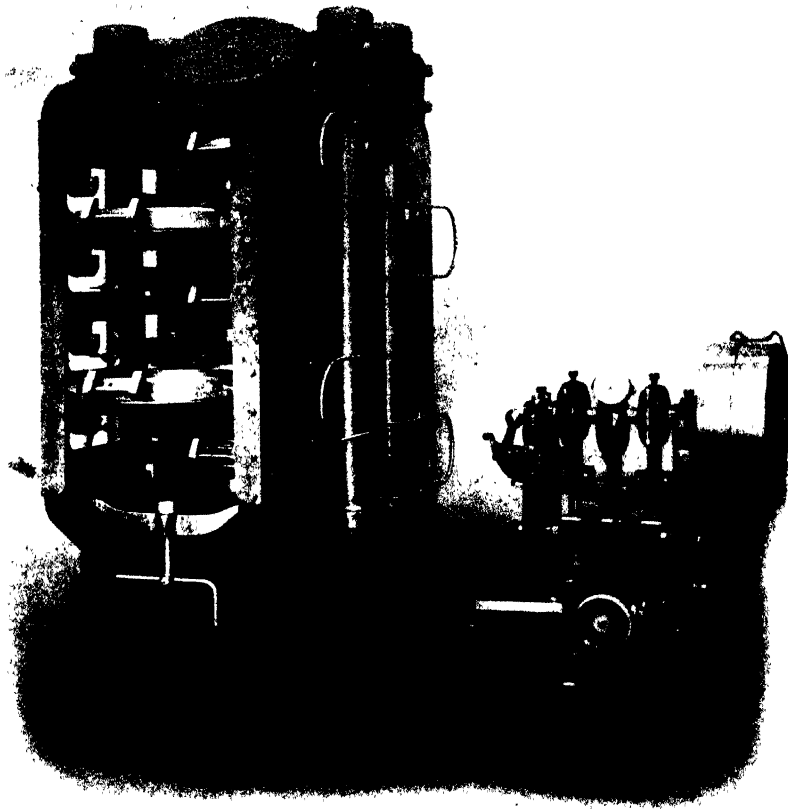


FIG. 10 —Ten-pot Hydraulic Cacao Butter Press, with Pump, etc. (See p. 170.)

*By permission of Messrs. Joseph Baker, Sons, and Perkins, Ltd.*

When the press is working, a piston fits telescopically into each cup, and the pressure, provided by a pump, forces the fat from the nibs, whilst the close fitting of one part in

the other and the inclusion of filter-pads prevent the escape of the cacao mass with the fat.

The fat, extracted by this method, will require filtering, if it is to be put upon the market, though such a course will be unnecessary, if the butter is used direct in the factory. If purification of the fat is necessary, it may be melted down and allowed to settle in a warm room, and the top portion may be drawn off, when all the sedimentary matter has settled to the bottom, or it may be filtered through a series of flannel bags.

For filtering large quantities, special machinery has been designed, in which the molten fat is passed first through a hair sieve to remove larger particles of cacao, etc., and then through hanging removable filter-bags, kept warm in a large tank. The tank receives the filtrate and is fitted with a gauge glass, so that the level of the fat in the tank can be ascertained.

Where large quantities of nibs are to be expressed, a battery of presses may be employed with advantage, and considerable economy and time can be effected by using an accumulator, attached to one large powerful pump. The pressure is maintained in the accumulator, which is connected to the pump on the one hand and with the presses on the other. The accumulator is coupled up with the presses through suitable inlet valves, and, as each press is furnished with a pressure gauge and the necessary valves, and as the actions of the pump and accumulator are automatic, the work is greatly simplified, and a constant pressure is maintained.

The fat or "cocoa butter" of commerce, more correctly termed "cacao butter," should be pale yellow and of a pleasant chocolate aroma and flavour. If too great heat has been used during its expression, the fat will be a dull grayish white, whilst, if the beans have been insufficiently roasted, the smell and taste of the butter will be "green" and unpleasant.

As in all cases of manufacture, technical skill and experience produce the best results, and, even with the highly efficient machinery at the command of the manufacturer, bad and inferior cocoas and cacao butters are placed on the market, through little or no attention being paid to the details of one or other of the many processes through which the cacao has to pass before being fit for consumption. •

#### **Cocoa Crushing Mill and Disintegrator.**

The cocoa, left in the press after the fat has been expressed, is removed and allowed to cool. It is then passed through a crushing mill, which consists of a pair of rollers fitted with teeth, and the mass is reduced to a fine powder. It may then be passed through another pair of smooth rollers, which complete the disintegration, and, after slight heating to reduce the cocoa to a light friable state, the powder is conveyed to the sifting machine.

#### **Cocoa Sifter.**

The sifter consists of a hopper into which the cocoa powder is delivered, and which, being furnished with a shake feed, delivers the cocoa into a rotating inclined hexagonal drum, covered with a fine-mesh silk.

The sifted cocoa falls into drawers, and the tailings, or portions which do not pass through the fine mesh, are carried over into a receptacle, placed at the end of the machine, whence they can be conveyed and returned to the crushing or disintegrating rollers to be re-ground.

This is one method of reducing the cocoa powder blocks, from the press, to a fine state of division ready for packing. When in New York, the author was shown, at the offices of Messrs. Lehmann Company Inc., the design of a complete automatic cocoa powder plant which he had seen previously in operation, working admirably. The plant consisted of a "melangeur" for crushing the cocoa



powder blocks and a Schutz O'Neil disintegrator for reducing the powder further, coupled together, and with a cooling and sifting plant from which the cocoa powder was delivered direct into trucks. The cooling arrangements were admirable and capable of easy regulation, and the cocoa powder which, though inevitably heated somewhat in the disintegration, after passing through the cooler had again acquired an excellent colour. The maintenance of colour in cocoa powder is no easy matter, if much friction occurs after it has become cold. It is for this reason that sieving arrangements prove very defective, and there is much to be said for wind-sifting, which separates the fine from the coarse particles to any degree, without bringing about dullness or whiteness in the cocoa powder. In the automatic plant of Messrs. Lehmann, the powder is sieved after cooling, but it does not seem to suffer much in colour during the process. The tailings are automatically carried back to the disintegrator by a worm conveyer, where they are again reduced and returned to the cooling chamber. The power required for driving the whole plant was supplied by one 25-h.p. and one 10-h.p. motor.

With regard to the temperature to which cocoa powder may be heated without deterioration of colour, it may be asserted that it has always been recognised, firstly, that, if cocoa powder is heated, it must be subsequently cooled if the colour is to be retained, and, secondly, that attrition of the cocoa powder, after cooling, is inclined to cause dullness and grayness. The Massachusetts Chocolate Company (Eng. Pat. 14,456, 1914) has perfected a system whereby the cocoa cakes are broken and heated to a certain temperature, 120° F. to 135° F., to the former if the cakes are warm from the press, to the latter if the cocoa cakes are cold. By these means, colour is imparted to the cocoa which it is necessary to "set." The "setting" of the colour is accomplished by cooling quickly and uniformly in a current of air,

at 40° F. to 45° F., which carries the powder and segregated particles from the breaking machine through chambers containing cooling pipes, where, by contact with the pipes and with one another, the cocoa is claimed to be reduced to a degree of fineness equivalent to 90 per cent. efficiency.

**"Soluble" Cocoa Powders.**

The term "soluble," as applied to treated cocoa powders, is undoubtedly a misnomer, for the property, attempted to be acquired by the manufacturer of so-called "soluble" cocoa, is in reality an emulsification, by means of which no deposit is formed at the bottom of a cup of prepared cocoa, even after prolonged standing. At first, this result was obtained by the incorporation of starchy matters with the cocoa powder, so that the starch, gelatinising on addition of boiling water, made a sticky and viscous mass which materially hindered the heavier particles from settling.

Hassall \* was a strong opponent to this method of rendering cocoa powder "soluble" and, in his book on "Adulteration of Food, etc.," condemns the practice not only by showing microscopic drawings of pure cocoa powders against those of cocoas with the addition of starch, but also by quoting lengthy cross-examinations of witnesses at the Parliamentary Commission, held to investigate the matter.

James Bell, in "The Chemistry of Foods," † says : "Most of the other preparations of cocoa, whether sold as soluble cocoa or chocolate, consist of mixtures of cocoa nibs with various substances ground together into a smooth paste. In the manufacture of soluble cocoa, arrowroot, sago or some other starch, and sugar, either dry or in the form of a syrup, are combined with the cocoa nib paste. The admixture of starch with the cocoa paste tends to mask the

\* Hassall, "Food : its Adulteration, etc.," 1876.

† J. Bell, "The Chemistry of Foods," 1887, 74.

presence of the fat and to render the cocoa more readily miscible with boiling water."

C. J. Van Houten was the first to attempt to treat cacao with chemicals, so as to render it more readily retained in suspension when mixed with water, without the addition of foreign starches.

By treating cacao with alkalis, such as potassium or sodium hydrates or carbonates, ammonia, or magnesium carbonate, the tissues of the kernel are partially disintegrated, and the material is capable of suspension in water or milk to a greater degree.

The slight saponification of the fat is also largely responsible for the formation of a satisfactory emulsion that will not allow the heavier portion of cocoa to deposit on standing.

The addition of alkalis to cacao has not met with unqualified approval, and there are many who, while readily acknowledging the superiority of the beverage resulting from the beans or cocoa so treated, are not prepared to admit that the inclusion of chemicals is not prejudicial to health.\* The nutritive value and digestibility of alkalis cocoas are discussed in a subsequent chapter.

The so-called "solubility" of modern cocoa powder is, however, a distinct advantage to the consumer, and, in the opinion of many, the treatment greatly enhances the flavour.

Bordas † has discussed the matter at some length and admits that, though French manufacturers, in general, do not add alkali salts for the purpose of producing "soluble" cocoa, the importation into France of foreign cocoa, so treated, has, of late years, reached very large proportions. In considering the desirability of addition of alkalis from the point of view of health, and the need of regulating by law

\* Vide discussion in Reports of Congress at Geneva and Paris, 1908, 1909.

† Bordas, *Ann. des Falsif.*, 1910, iii., 61—70.

the quantity added, Bordas points out that natural cacao mass contains from 2.46 per cent. to 3.05 per cent. of potash, estimated as  $K_2O$  and calculated on the dry fat-free substance, whilst samples of "soluble" cocoas contain from 4.82 per cent. to 6.41 per cent. of the same material. As there is no direct evidence of injury done to the health by the consumption of such cacaos, Bordas concludes that, provided the quantity of potash is not allowed to become excessive, there is no harm in treating the cacao with alkalis.

The same author remarks that Belgium, Italy and Switzerland prohibit the sale of cocoa containing more than 3 per cent. of alkaline carbonate, and that, in Roumania, the limit is fixed at 2 per cent. The United States prohibited the sale of cocoa containing any added alkali, and the same regulation obtained in Austria. This statement is, however, not strictly accurate, and the regulations obtaining in different countries, on this point, are discussed in a later place.

The invention of C. J. Van Houten, in 1828, marks the first step in alkalisied cocoa powders, and is usually called the "Dutch process." Firstly, the extremely fine comminution of the cocoa powder renders the deposition of the heavier particles from a cup of cocoa more difficult, and, secondly, the formation of an emulsion by the addition of alkali helps towards the same end. The arguments that have raged against the use of alkali from the hygiene point of view depend, on the one hand, upon the rabid dieteticians and, on the other hand, upon the more irrational chemists.

By treatment with potash, the ratio of soluble to insoluble ash, originally present in the cacao as about 46 : 54, is increased in favour of the soluble ash to about 80 : 20. From this point of view, the treatment with alkali is a distinct "solubilising" process. Treatment by steam or

ammonia will not bring about this increase of soluble ash, but, on the other hand, these operations will not increase the ash figures at all, the only point in their favour.

Potash treatment improves the flavour and odour of cocoa powder and darkens the colour, the latter effect being due, probably, to the oxidation of the alkali salts of tannic acid which is to be found in untreated cacao. Further, the treated cocoa powders are not alkaline in reaction themselves, whilst untreated cocoa powders are acid, due both to acid salts of phosphoric acid and to tannic and other free organic acids. The effect, then, of the addition of alkali is to neutralise acidity, with the formation of potassium salts of phosphoric, tannic and other acids which, on incineration, will yield an alkaline ash.

Owing to the weakness of its basicity compared with potash, ammonia is far less efficient, and the difficulty of removing the objectionable ammoniacal flavour is obvious.

If too much alkali is added, clearly, the cocoa powders will show an alkaline reaction, and, on calculation, it is found that from 2—3 per cent. of added alkali, as  $K_2CO_3$ , is sufficient to bring about neutrality. Analysis of Van Houten's product shows from 2.05—3.05 per cent. calculated as  $K_2CO_3$  on the dry powder.

The usual mistake of manufacturers, commencing to make "soluble" cocoa powders, is that of adding too much or too strong alkaline solutions. It is necessary to add just sufficient alkali to neutralise the organic acids, and of just sufficient strength to open effectually the pores of the tissue of the bean. Two parts of potash to 100 parts of the bean should be quite enough to accomplish these two objects.

Treatment with steam is effective in rendering cocoa "soluble," but for another reason. Cacao contains about 12 per cent. of starch, calculated on the fat-free nib, and the action of steam is to cause the starch grains to swell, when they readily take up water and become more or less soluble

on the addition of the liquor when the beverage is prepared. The cocoa becomes viscous by this treatment, though, in reality, it should not be different to the cocoa beverage prepared, in the proper manner, by the addition of boiling milk or water and by a subsequent further boiling, when the starch grains will swell, producing a viscous beverage without preliminary treatment of the powder.

### **Methods of Application of Alkali.**

The treatment of the cacao with alkali may be accomplished with water and heat, with or without pressure, with the alkalis potassium, sodium or magnesium carbonates, usually used in Holland, or with solutions of ammonia or ammonium carbonate, often employed in Germany. The hydroxides of sodium and potassium are also used in the production of "soluble" cocoas.

The alkalis may be applied before, at the time of, or after roasting, before the pressing, and, consequently, after milling, or after the pressing.

There is, therefore, plenty of scope for the manufacturer, and, if one method does not give the required result, another may be tried, for the treatment with any one alkali, at any one stage, does not produce a cocoa similar in flavour or qualities to that treated at a different stage in its manufacture with a different alkali.

If the cacao is to be treated before roasting, the beans may be subjected to the influence of steam and ammonia, which is the method suggested by Staehle in a German patent. In this process, he suggests that the operation be carried out below 100° C., and the beans subsequently roasted at 130° C., when the ammonia, being volatile, is driven off, and the aroma of the cacao is developed.

Neumann, in a French patent obtained in 1909, washes the beans in a solution of alkali at 50° C. The cacao is removed, and the temperature gradually raised to 120° C.,

when the beans are roasted. In subsequent processes, the shells are removed, and the nibs ground to a paste, when, before or after expressing the fat, the mass is again gradually heated to 100° C. and ground in a "melangeur" from one to twenty-four hours. By these means, he claims to obtain a "soluble" cocoa powder of fine flavour.

- The beans may be treated with alkali after a slight roasting; thus, in one process, the beans, after being gently heated and shelled, are sprinkled with a solution of potassium carbonate containing 1·5 per cent. to 3 per cent. of the alkali. If too much alkali has been added so as to give an unpleasant taste to the cacao mass, a solution of tartaric acid is added to neutralise the effect. The mass is now fully roasted over a gentle fire in a clean roaster, and great care has to be taken to prevent the treated cacao from burning, as the mixture, clinging to the revolving roaster, is likely to become superheated where in contact with the metal. Subsequently, the dried cacao is expressed.

Neumann, in an English patent, 1910, adopts a somewhat similar process. By this method, the beans are completely roasted and husked, and the nibs, reduced to a coarse meal, are slightly pressed, to get rid of some of the fat. The partially defatted cacao is then treated with alkali and again pressed.

Pieper, in a German patent of 1893, besides providing a specification, gives scientific arguments in favour of his process. Briefly put, the process and arguments in its favour are as follow :—The raw beans are treated with an alkaline solution to neutralise the free acids, of such a strength that neutrality is not exceeded. The beans are allowed to ferment, somewhat, for twelve to forty-eight hours, dried and roasted. The fermentation process in a neutral or slightly alkaline solution is said to render the albuminoids more easily digestible, whilst the roasting converts the starch into dextrins after hydration. The fermentation is

claimed also to convert the blue-violet or slate-gray colour, characteristic of some cacaos of lower quality, into the red-brown colour, of the better kinds of cacao, which is so much sought by the chocolate manufacturers. Pieper's process consists in adding, first of all, about ten parts of water, at  $20^{\circ}$ — $22^{\circ}$  C., to 100 parts of cacao and in neutralising the free acids (determined chemically) by the correct amount of alkali. The fermentation is allowed to take place in wooden vessels, and a rise in temperature is noticed after three to six hours. During fermentation, the beans rise in temperature to  $27^{\circ}$ — $29^{\circ}$  C., which should be maintained for about forty-eight hours. The neutralisation of the acidity is claimed to prevent coagulation of the milk, subsequently added to cocoa powder prepared in this manner, and to activate the enzymes contained in the cocoa.

Some of the claims are obviously extravagant, though the author has seen good cocoa of the "soluble" sort prepared in this way. It is doubtful, however, whether the efficiency of Pieper's process is greater than the simple processes of adding alkalis direct to the cacao mass or cocoa powder, provided that a chemical check is kept upon the amount of alkali added, so that the organic acids are only just neutralised.

The best results that we have obtained are from a process substantially as follows:—The beans are half roasted, crushed, husked and winnowed, and placed in wooden troughs or tubs containing some 100 lbs. of nibs. A solution of one part of potash in five parts of water is worked well into the nibs, in quantities sufficient to neutralise the organic acids in the nibs, previously determined in the laboratory. The troughs or tubs are covered with boards or damp cloths and conveyed to a room at about  $30^{\circ}$  C., where they rest for twenty-four to twenty-eight hours, to undergo a light fermentation. The nibs are then roasted fully in a roaster from which it is possible to remove the moisture



given off, the greatest care being taken that the cacao, now deprived of its protective coating and husk, does not burn. The subsequent treatment, milling and pressing, is the same as for ordinary cocoa powder. The nibs may be dried before the second roasting by placing them in shallow trays in the hot room or by other convenient method.

- In yet another process, magnesium carbonate, or, better, a strong solution of caustic potash (90—95 per cent.), is mixed in, or sprayed on, to the cacao paste from fully roasted beans, and, after thorough mixing, the treated mass is pressed in the usual way. Another process is described as follows :—“ To 100 lbs. of cacao paste are added 2 to 3 lbs. of potash, dissolved in 1 to 1½ gallons of water, and the whole is thoroughly kneaded in a dough mixer or apparatus of similar construction. If ammonium carbonate is used instead of potash, ½ to 3 lbs. are used for the same bulk of cacao paste with the same volume of water. The treated cacao is then placed in the hydraulic press, and the fat expressed.”

The application of alkali may be made, after the fat has been expressed, in the form of a liquid spray, and, in such cases, potassium carbonate, and not ammonia or ammonium carbonate, solutions should be used, as it is extremely difficult to rid the treated cacao of the ammoniacal smell, if no high temperature is employed subsequent to their application. In all cases where aqueous solutions are used, care should be taken to free the cacao mass or powder from water, as, without such precaution, the resulting powder is liable to turn mouldy.

There are other processes for making “soluble” cocoa powders, which do not entail the use of alkalis, but, as these depend largely on treating the cacao with boiling sugar, it has been thought desirable to defer their discussion until a later chapter which deals with cocoa preparations and mixtures of cocoa with sugar, milk powder, etc.

There are, also, many mechanical processes for rendering

cocoa "soluble." To quote one example (English Patent 14,758, 1913), Bergmuller recommends the treatment of the cacao mass at 60°—65° C. in a centrifugal machine for four to five hours when, it is claimed, the cacao butter is brought to the surface of the particles, whence it can be more readily and completely expressed in a hydraulic press, under a pressure of 600 atmospheres at about 88° C. ; or the cacao mass may be treated with one litre of water per 100 kilos of cacao previously to putting the mass into the press, the water being absorbed by the hygroscopic particles of cocoa. Such devices, however, whilst probably facilitating the removal of a large quantity of fat, do not make for a good cocoa powder, but rather for a large yield of cacao butter.

Owing to the considerable demand for "soluble" cocoas in recent years, it has been found necessary to increase the efficiency of existing machinery and to create new, in order to accelerate the process.

The following plant is necessary to manufacture about 1 ton of pure "soluble" cocoa essence per ten-hour day. The machines should be obtained only from the best engineers in the industry :—

One machine for cleaning and sorting cacao beans.

Two cacao-roasting machines.

One exhaust fan, with two cooling trolleys.

One cacao-crushing, cleaning, hulling and separating machine.

One germ-separating machine.

Three triple cacao mills.

Three hydraulic cacao butter presses, with one accumulator.

Two "Universal," or similar mixers.

One automatic pulverising plant, consisting of :—

One breaking machine.                      One pulveriser.

One sifting machine.

Total h.-p. required, about 20.

## CHAPTER XIV

### ANALYSES OF COCOA POWDERS, "SOLUBLE," DIETETIC, PROPRIETARY, ETC.

ACCORDING to the processes to which the beans have been submitted, described in the preceding chapters, so will the cocoa powders, prepared from them, show, on analysis, varying fat and ash contents and differing values for the alkalinity of the mineral matter.

Thus, a powder which has been heavily expressed may show as low a fat-content as 10 per cent., rising to 40 per cent., if the pressure of the extracting press has been light.

Or, again, the application of alkalis, such as potash and potassium, sodium or magnesium carbonates, will be more apparent in the analyses of the finished cocoa powder, if it has been made to the nibs, cacao paste or powder, than in cases where the beans have been treated still in their shells.

The volatile alkalis, ammonia or ammonium carbonate, will not show on analysis, as it is improbable that they form any stable compounds with the component parts of the cacao, and they are unlikely to appear in the ash. On the other hand, a cocoa powder showing a very high percentage of nitrogen may be suspected of being treated with, and not freed from, ammonia, though it is unlikely that any ammonia or its compounds could be present without serious detriment to the flavour.

Stutzer,\* however, who has made a close study of the effects of the different processes of manufacture on the resulting analyses, claims to be able to detect cocoas pre-

\* Stutzer, *Zeitsch. angew. Chem.*, 1891.

pared by treatment with ammonia. His results are given in Table XXXIV.

TABLE XXXIV.—*Analyses of Cocoa Powders, showing influence of methods of manufacture on the figures obtained.*

	I	II.	III	IV. •
Water . . . . .	4.30	3.83	6.56	5.41
Fibre . . . . .	3.36	37.48	39.99	36.06
Nitrogen-free extract . . . . .	38.62	19.88	20.93	19.25
Total nitrogenous substances * . . . . .	20.84	30.51	27.34	33.85
Fat . . . . .	27.83	8.30	5.18	5.43
Ash . . . . .	5.05			
	100.00	100.00	100.00	100.00
*Containing total nitrogen . . . . .	3.68	3.30	3.95	3.57
Composed of:—				
Theobromine . . . . .	1.92	1.73	1.98	1.80
Ammonia . . . . .	0.06	0.08	0.46	0.33
Amido-compounds . . . . .	1.43	1.25	0.31	1.31
Digestible albumen . . . . .	10.25	7.68	10.50	7.81
Indigestible nitrogenous substances . . . . .	7.18	9.19	7.68	8.00
Containing nitrogen . . . . .	1.15	1.47	1.23	1.28
Proportion of total nitrogen indigestible . . . . .	31.2	44.5	31.2	35.8
(Containing— Total $P_2O_5$ . . . . .	1.85	2.52	2.14	2.05
$P_2O_5$ soluble in water . . . . .	1.43	0.50	0.74	0.77
Ratio of total $P_2O_5$ to soluble . . . . .	100 : 77	100 : 19	100 : 34	100 : 37
Ash soluble in water . . . . .	3.76	4.76	2.82	2.76
Ratio of total ash to soluble . . . . .	100 : 74	100 : 57	100 : 54	100 : 49

I. was composed of forty parts Arriba, forty parts Machala and twenty parts Bahia beans, and was manufactured by Wittekop and Company without the use of chemicals.

II. was a sample of well-known cocoa, manufactured in Holland, with the addition of potash.

III. and IV. were German cocoas prepared, according to Stutzer's opinion, by the application of ammonia.

An interesting series of analyses of cocoa powders, made by Hughes,† at the request of the Planters' Association of

† Hughes, "Analyses of Samples of Cacao," 1899.

Ceylon, is given in Table XXXV. which, besides showing the percentage chemical composition, indicates the mechanical condition of the cocoa, a matter of great importance both to the consumer and manufacturer.

TABLE XXXV.—*Analyses of Cocoa Powders.*

	Cadbury's.	Fry's.	Van Houten's.	Rowntree's.	Tibbles'.	Epps's.
Price per lb. (1899) .	2s. 4d.	2s. 1d.	3s. 0d.	2s. 9d.	2s. 9d.	1s. 2d.
Water (loss at 100° C.) .	3.99	4.30	2.94	3.82	5.04	4.58
Fat (cacao butter) .	25.01	30.70	30.10	32.50	27.10	26.40
Albuminoids and theobromine . . . .	22.15	20.38	21.04	19.30	17.53	7.27
Mucilage, gum, etc. .	9.70	10.64	10.03	9.04	11.86	11.82
Sugar, glucose, and dextrin . . . .	4.10	1.36	0.97	1.76	2.34	18.53
Starch and digestible fibre . . . .	25.91	23.76	21.44	21.83	27.10	28.45
Indigestible fibre . .	4.31	4.46	4.26	3.83	5.33	1.40
Ash or mineral matter .	5.06	4.40	8.62	7.92	6.50	1.50
	100.00	100.00	100.00	100.00	100.00	100.00
Melting point of fat .	28.9° C.	29.4° C.	28.9° C.	29.4° C.	26.7° C.	28.3° C.
Containing nitrogen .	3.50	3.22	3.42	3.05	1.77	1.55
Containing potash . .	1.66	1.56	4.49	4.24	2.08	0.54
<i>Mechanical condition</i>						
Powder passed through sieve (10,000 holes to the square inch) .	72.59	81.78	57.05	62.51	76.35	42.74
Residue left on sieve .	27.41	18.22	42.95	37.49	23.65	57.26
	100.00	100.00	100.00	100.00	100.00	100.00

In considering these figures, the author points out that, in Cadbury's and Fry's cocoas, about two-thirds of the original fat of the cacao had been removed, but that they were otherwise untreated. Van Houten's and Rowntree's cocoas were taken as samples of cocoa powders from which two-thirds of the original fat of the cacao had been removed, but which had been treated with potash salts.

To both Tibbles' and Epps's cocoa powders, other materials, such as starch and sugar, had been added, and, consequently, in these powders, the chemical components of cacao were found in reduced proportions.

In the four samples, Cadbury's, Fry's, Van Houten's and Rowntree's, no foreign starch or sugar was present, the amount of reducing matter and starch, found, being due to that naturally existing in cacao.

Analyses of various commercial cocoa powders are published from time to time, and, in Table XXXVI., will be found a few in which the alkalinity of the ash (the number of ccs. of  $\frac{N}{10}$  acid required to neutralise the ash from 2 grms. of sample) has been converted into figures to conform with that value, as usually expressed.

TABLE XXXVI.—*Analyses of some well-known Brands of Commercial Cocoa Powders.*

Constituents.	Fry's Cocoa Extract.		Van Houten's Cocoa.			Rowntree's Extract of Cocoa.		Baker's Dutch Cocoa.		Bens- dorp's Pure Royal Dutch Cocoa.	Cad- bury's Cocoa Essence.
	I.	II.	I.	II.	III.	I.	II.	I.	II.	III.	II.
Moisture . . .	—	4.33	—	4.53	—	—	4.05	—	—	4.50	4.00
Ash: { Total . . .	4.24	4.28	8.64	8.19	7.96	8.48	7.70	6.06	6.10	6.60	4.70
Alkalinity as $K_2O$ . . .	1.33	—	3.68	—	1.81	3.82	—	2.21	1.24	—	—
Fat . . .	30.95	31.16	29.80	29.78	29.66	27.56	30.82	31.48	27.07	33.06	27.58
Extractive soluble in water . . .	—	5.26	—	9.88	—	—	7.48	—	—	8.52	6.48
Theobromine . . .	—	1.36	—	0.69	2.00	—	1.08	—	1.90	0.88	0.70
Starch . . .	—	16.07	—	21.26	—	—	—	—	—	11.33	21.05
Albumen . . .	—	12.78	—	17.03	—	—	15.22	—	—	11.41	13.58
Fibre . . .	3.80	—	4.38	—	—	4.42	—	3.76	—	—	—

I. E. E. Ewell, *Bull. No. 13, U.S. Dept. of Agriculture.*

II. Florence Yaple, "*Chem. Zeit.*," 1895, Rep. 21, 240.

III. C. Girard, *Internat. Cong. App. Chem.*, 1909, Sect. VIII. C. 185.

In considering the figures for the alkalinity of the ash in the above table, it should be noticed that Girard's values are made on the ash soluble in water only, whilst Ewell's figures, being on the total ash, will include the calcium and magnesium carbonates, which would not be found among the soluble portion.

Girard, whose work, already quoted, was primarily

undertaken for the purpose of studying the presence of oxalic acid and the estimation of the same, gives some tables showing the analyses of various cocoa powders of commerce and the composition of their ash. His results are so particularly valuable and enlightening that it has been thought desirable to include them here in full.

TABLE XXXVII.—*Analyses of some Commercial Cocoa Powders.*

	Van Houten's	De Jong's	Es- brique & Amster- dam	Bens- dorp	Such- ard	Bloo- ker	Conté	Menet	Mor- cuil	Choco- latrie Ou- vrière.
Ash	7.96	7.22	6.98	6.70	6.36	6.10	6.36	4.84	4.46	4.18
.. Insoluble	15.57	16.61	19.19	22.38	22.01	25.90	21.06	55.77	58.25	60.28
.. Soluble	84.43	83.39	80.81	77.62	77.99	74.10	78.94	44.23	41.75	39.72
Alkalinity of soluble ash as K <sub>2</sub> O	1.81	1.08	1.47	1.27	1.43	1.24	1.38	0.47	0.49	0.40
Sucrose	<i>Nil</i>	<i>Nil</i>	<i>Nil</i>	<i>Nil</i>	<i>Nil</i>	<i>Nil</i>	<i>Nil</i>	<i>Nil</i>	<i>Nil</i>	<i>Nil</i>
Reducing matters	Traces	Traces	Traces	Traces	Traces	Traces	Traces	Traces	Traces	Traces
Fatty matter	29.66	30.27	25.07	29.77	31.67	27.07	29.18	23.27	34.64	36.46
Melting-point of fat	31.0	29.8	30.4	31.2	30.2	30.8	30.4	30.4	30.6	30.4
Jeau refractor value of fat	20	17.5	18.5	19	19	19	19	18	18.5	18.5
Saponification value of fat	192.7	193.6	192.3	193.0	192.8	192.6	193.6	191.0	192.2	192.1
Iodine value of fat	35.4	39.4	38.0	36.3	37.4	34.9	37.6	37.4	36.7	39.0
Theobromine	2.00	1.88	2.06	2.05	1.94	1.90	2.16	2.24	1.84	1.04
Oxalic acid	0.406	0.630	0.630	0.567	0.566	0.504	0.630	0.651	0.536	0.504

Leaving for one moment the ash and its values, the significance of the other figures is worthy of brief consideration at this point.

*Sucrose.*—The absence of sucrose in the powders shows that no addition of sugar has been made and that the beans were well fermented (*vide* Chapter IX., Table XIV., *Sucrose*).

*Reducing Matters.*—The presence of only traces of reducing matters shows that the beans have been well fermented, for cacao kernels have been shown to contain 1.5 per cent. of glucose and other reducing matters, when merely dried, and up to 1.0 per cent., if slightly fermented. If the beans have been thoroughly fermented, there should be only traces of reducing matters.

*Fatty Matter.*—The lowest percentage of fat is seen in Menier cocoa, and the highest in "Chocolaterie Ouvrière." The percentage of fat has been shown to depend upon the degree of expression to which the roasted nibs have been subjected.

The melting-point, refractometer, saponification and iodine values of the fat fall within the limits of those obtained for pure cacao butter, and, consequently, in the powders analysed, no addition of, or adulteration with, foreign fat has been made. The interpretations of these values and the methods for their estimation will be discussed fully in the third part of this book.

*Theobromine and Oxalic Acid.*—The percentages of both these constituents of cacao are mainly of academic interest, though, if the beans had been too highly roasted, the quantity of theobromine, to which is due the stimulating nature of cocoa preparations, would have been lower than 1.0 per cent. of the powder.

In considering the ash percentage and values, it will be desirable to tabulate the chemical composition of the mineral matter or ash, obtained from the different cocoa powders. The mineral matter has been completely analysed by Girard, and his results will be found in Table XXXVIII.

*Ash.*—In the first seven powders, the sum of the potash and soda values equals some 50 per cent. of the total ash, though, only in the first, does the quantity of soda amount to any appreciable quantity. In this case, the powder must certainly have been treated with sodium hydroxide or carbonate, since the ash of normal, untreated powders contains some 30 per cent. of potash ( $K_2O$ ), as will be seen in the last three cocoas which have been prepared without the addition of alkali, and which do not themselves contain more than  $2\frac{1}{2}$  per cent. of soda.

In the cocoas 2 to 6 inclusive, it is safe to say that either potassium hydroxide or carbonate had been used to render



TABLE XXXVIII.—Analyses of Ash obtained from Commercial Cocoa Powders.

	Van Houten's.	Dejong's.	Fabrique & Amster- dam.	Benadorp	Suchard.	Bloeker.	Conté	Menet.	Morcuil.	Choco- latrie Ouvrière.
<i>Ash soluble in water :</i>										
Potash as $K_2O$ . . .	22.71	52.07	47.68	43.31	42.41	43.15	46.77	• 22.68	24.23	19.73
Soda as $Na_2O$ . . .	22.32	0.71	1.03	0.61	0.85	0.61	0.86	2.44	0.30	2.40
Sulphuric anhydride, $SO_3$ . . .	1.84	1.59	3.20	2.59	2.29	2.00	2.47	2.38	2.60	2.30
Chlorine, Cl . . .	0.96	0.45	0.58	0.45	0.57	0.77	0.19	0.61	0.18	0.67
Phosphoric anhydride, $P_2O_5$ . . .	21.54	17.08	17.46	17.40	19.82	18.26	19.23	7.64	6.98	8.30
Carbonic anhydride, $CO_2$ . . .	14.39	12.46	10.96	10.84	11.13	9.73	10.00	7.85	6.66	6.60
Lime and magnesia, $CaO$ and $MgO$ . . .	Traces.	Traces.	Traces.	Traces.	Traces.	Traces.	Traces.	Traces.	Traces.	Traces.
<i>Ash insoluble in water :</i>										
Potash as $K_2O$ . . .	0.26	0.26	Traces.	0.54	0.31	0.83	0.93	6.85	10.68	11.04
Soda as $Na_2O$ . . .	—	—	—	—	—	—	—	—	—	—
Lime, $CaO$ . . .	1.25	1.93	2.39	3.35	2.26	1.78	3.21	3.39	3.42	3.26
Magnesia, $MgO$ . . .	10.33	9.84	11.17	12.09	11.96	11.99	11.67	16.57	15.19	16.32
Iron oxide, $Fe_2O_3$ . . .	0.83	0.78	0.61	0.52	0.50	0.98	0.61	0.49	0.50	0.76
Alumina, $Al_2O_3$ . . .	0.15	0.31	0.19	0.15	0.18	0.67	0.24	0.08	0.09	0.37
Silica, $SiO_2$ . . .	0.77	0.69	1.56	0.49	0.85	1.38	0.53	1.67	1.91	2.06
Phosphoric anhydride, $P_2O_5$ . . .	2.59	1.59	3.11	5.31	5.72	6.13	2.38	24.77	20.63	22.36
Carbonic anhydride, $CO_2$ . . .	0.28	0.24	0.19	0.45	1.28	0.19	0.95	2.50	6.48	3.98
Sulphuric anhydride, $SO_3$ . . .	Traces.	Traces.	Traces.	Traces.	Traces.	Traces.	Traces.	Appreci- able traces.	Appreci- able traces.	Appreci- able traces.
	100.22	100.10	100.13	100.10	100.13	100.17	100.04	100.13	100.05	100.15

them "soluble" and that their addition, as also of the soda in the first, had succeeded in fixing the phosphoric, carbonic, and sulphuric acids, originally present, as insoluble compounds. That is to say, the alkali, added, attracts to itself the acid portions of the powders forming soluble salts. with the result that, on analysis, those cocoas treated with alkali are found to contain soluble ash with greatest proportions of potassium, sodium, or magnesium salts of phosphoric, carbonic and sulphuric acids, according to the alkali used.

In the last three cocoa powders, the comparatively low percentages of potash, soda, and phosphoric and carbonic acids in the soluble ash tend to show not only that the cocoas were prepared without the addition of alkali, but also that the remainder of these components, not to be found in the soluble ash, are combined in some probably complex form which will not split up without the presence of excess of alkali, but which will, consequently, figure in the ash insoluble in water.

Not without interest are some results, recorded in the *Lancet* of January, 1905, to which we have occasion to refer again in a later chapter. The authors of this research endeavoured to ascertain the effect of alkalis on cocoa powder upon the digestion, or rather to obtain the digestibility and nutrition values of different cocoa powders. They showed that the cocoas, if treated, were in no sense "alkaline," though possibly "alkalised." The statement appears in this article that, "of the eleven cocoas purchased in the open market we have not found a single one to contain anything in the nature of an alkali. They were either acid in reaction or perfectly neutral as regards alkali. By alkali we mean either the carbonates or hydrates (caustic) of the so-called alkaline metals (potassium or sodium). Moreover, in no instance was the slightest trace of soap found, formed by the action of the alkali on the cocoa fat." It has

always been the contention of the author that properly alkalisied cocoa powders should not be alkaline in reaction, yet, in the face of the table published by the writers in the *Lancet*, their subsequent statement seems unwarranted and uncalled for :—" Certainly as regards the well-known manu-

TABLE XXXIX.—*Ash and Alkalinity of some Cocoa Powders.*

Cocoa.	Total mineral matter (ash)	Alkalinity of ash reckoned as potash.	Alkali in cocoa.
1. Bendsdorp's Soluble, II . . . . .	7.10	2.35	None
2. Barry's Essence, I . . . . .	4.60	1.08	..
3. Van Houten's Soluble Cocoa, II . . . . .	8.80	3.14	..
4. Epps's Cocoa Essence, I. . . . .	3.90	0.75	..
5. Fry's Concentrated Cocoa, I . . . . .	5.50	1.97	..
6. Cadbury's Cocoa Essence, I. . . . .	4.00	0.65	..
7. Mazawattee Cocoa, II . . . . .	5.50	2.06	..
8. Schweitzer's Cocountina, II. . . . .	5.70	2.02	..
9. Suchard's Soluble Cocoa, II. . . . .	6.40	3.55	..
10. Rowntree's Elect Cocoa, II. . . . .	7.40	3.14	..
11. Loose Cocoa, II . . . . .	7.70	2.96	..

1. Slightly alkaline potassium salts, but neutral as regards free alkali.
2. Slightly acid reaction.
3. Slightly alkaline reaction due not to alkali, but to organic salts of potassium, and phosphates; otherwise perfectly neutral.
4. Acid reaction.
5. Acid reaction.
6. Acid reaction.
7. Neutral reaction.
8. Neutral reaction.
9. Neutral reaction.
10. Neutral, rich in organic salts of potassium, including phosphates.
11. Neutral, rich in organic salts of potassium, including phosphates.

facturers of cocoa which is sold in this country, whether English or Dutch, there is here no evidence whatever of the use of alkalis in the preparation of cocoa. There is abundant evidence, on the other hand, showing that the natural organic salts of the cocoa have been materially increased as regards both some of the English and Dutch manufactured

cocoas." The two arguments, unfortunately, are in conflict, especially if the usual methods adopted in the preparation of ordinary and "soluble" cocoa powders are known. We will, however, leave it at that, and give the above table and comments which appear among many others in the *Lancet* of January, 1905.

The natural organic salts, mentioned, consist of tartrates, malates and citrates of potassium, which all give slightly alkaline reaction and yield potassium carbonate, on incineration. Alkaline potassium phosphate was also found in several of the cocoas examined. "Of such cocoas (Class II. in the table shown) it would be just as true to say that they contain alkali as it would be of wood because on burning it yields potash or potassium carbonate in the ash"—a truly unscientific assumption.

In France, in 1910, a decree forbade the application of the word "pure" to any cocoa powders other than those obtained by pulverising and partially defatting cacao mass. The word "pure" could, however, be applied to cocoa powders rendered "soluble" without addition of chemical substances, though the decree did not look upon as adulteration the use of alkalis or alkaline carbonates to cocoa, destined for preparation of cocoa powder, provided that the quantity added did not exceed 5.75 grms., or an equivalent quantity of another alkaline carbonate, per 100 grms. of cocoa, calculated as free from moisture and fat. The cocoa powder, under such circumstances, had to show a slightly alkaline reaction. Later, the official method \* of analysis of chocolate and cocoa included a statement to the effect that the percentage of alkalinity, expressed as potassium carbonate on the dry fat-free cocoa, must not exceed 2.75 per cent. in those products which had not received any addition of alkali, and that "soluble" cocoas, prepared according to article 18 of the decree, already quoted, must not

\* *Ann. des Falsif.*, August, 1911, 418.

exceed  $5.75 + 2.75 = 8.50$  per cent. of potassium carbonate.

At the end of 1915, after the examination of three cocoa powders, purchased in a large store in Paris, the "Laboratoire Central du Ministère d'Agriculture" announced that the statement on the label, "Cacao en poudre garanti pur," could not be applied to these cocoas, since they showed the following analyses, later confirmed by independent analysts:—

	A.	B.	C.
Moisture per cent.	3.06	3.90	5.40
Fat per cent.	32.70	31.20	31.30
Ash per cent.	4.58	5.30	5.20
Alkalinity of ash, as $K_2CO_3$ , per cent. of dry defatted cocoa.	3.50	3.10	3.10

It was clear that, had the cocoa been "alkalised" for any particular purpose of benefit to the manufacturer, the amount of alkali in the ash would have been higher than in normal cocoa, calculated on the same basis, a point overlooked by the writer in the *Lancet*. It was from this standpoint that Arpin\* carried out his research, and that Rocques, at the same time, devised his method for estimation of alkalinity. Arpin found the following results from raw beans, after the husk and germs had been removed:—

	Trinidad	Mada- gascar	Caru- pano.	Ibarrá.	Chuao.	Puerto Cabello.
Moisture per cent.	6.36	6.37	7.23	7.21	7.27	7.91
Fat per cent.	47.60	48.40	42.70	43.80	43.80	46.90
Ash per cent.	2.95	3.16	2.90	2.96	3.63	3.16
Alkalinity of ash, as $K_2CO_3$ , per cent. of dry and defatted cocoa.	3.10	3.53	2.92	3.10	3.59	2.96

\* Arpin, *Ann. des Falsif.*, January, February, 1917, xcix., c. 10.

It was thus established that raw untreated cacao had an ash showing an alkalinity in excess of that allowed by the French decree of 1910.

In the same research, Arpin gives the following table, in which the present author has also included the figures for germ:—

	Raw husk of					Roasted Trinidad germ
	Accra	Bahia	Caru- pano	Chuao	Mada- gascar	
Moisture per cent. . . . .	12.28	13.24	9.52	8.60	11.93	4.40
Ash per cent . . . . .	6.79	6.60	7.43	11.60	5.52	6.25
Alkalinity of raw shells . . .	5.24	4.23	4.69	4.56	4.97	—
Alkalinity of dried shells . .	5.97	4.87	5.18	4.99	5.64	—
Alkalinity of roasted germ . .	—	—	—	—	—	4.13
Fat per cent . . . . .	—	—	—	—	—	4.44

After further examination, in which the good faith of the manufacturers of the cocoa powders was established, the following points were formulated by Arpin:—

(1) That cacao husks show a variable moisture-content and that the driest are those which might be suspected of having been treated with an alkaline solution.

(2) That the ash-content varies from 5.52 per cent. to 11.60 per cent. (in the specimens of husk examined) without the alkalinity increasing proportionally. The ash is composed of inert bodies, sand, earth, etc., which increase the mineral-content; that those varieties examined, which gave 7.43 per cent. and 11.10 per cent. of ash, respectively, had been "earthed" or "clayed."

(3) That the alkalinity of the husks of cacao is the same within limits of 1 per cent.

(4) That the husks of Accra beans are most alkaline (5.97 per cent. calculated on the dry husk), whilst the cacao itself does not reach the figure 2.75 per cent.

(5) That the husks of Bahia cacao (termed non-alkaline) are almost identical in alkalinity (4.87 per cent.) to that of

the husks of Carupano (5.18 per cent.) and Chuao (4.99 per cent.) which are called alkaline cacaos.

(6) Finally, that the husks of Madagascar cacao, though termed an alkaline cacao, are less high in alkalinity than those of Accra which is a non-alkaline cacao.

Generally speaking, these figures show that husks of cacao, showing greater alkalinity than 2.75 per cent., have not necessarily been treated with an alkaline solution or with a solution capable of giving an alkali on incineration.

The present author fears that a fundamental error appears in all these figures, owing to the fact that Arpin has taken for granted that Chuao and Madagascar cacaos had been treated with alkaline solutions and that the Accra, Bahia and Carupano beans had not. In any case, the conclusion, reached, that the small quantity of husk finding its way into the cocoa powder, accidentally or through carelessness on the part of the manufacturer, is without appreciable effect upon the alkalinity of the finished product, seems justified.

#### **Dietetic, Proprietary, and other Cocoa and Chocolate Powders.**

Cocoa powders should contain the roasted, partially defatted kernels of cacao beans only. The chemical composition of such cocoa powders will depend upon the amount of expression to which the nibs have been subjected. If 30 per cent. of fat has been expressed, which is usually the highest limit in practice, the resulting cocoa powder will show 25—35 per cent. fat, 4—5 per cent. ash, 3—5 per cent. fibre, 12—17 per cent. nitrogenous matter as albuminoids, 11—20 per cent. starch, and 4—5 per cent. moisture.

"Soluble" cocoa powders, treated with alkali, should consist of the roasted, partially defatted kernels of cacao beans, with the addition of sodium or potassium hydroxide or carbonate, or magnesium carbonate, and, if the nibs have

30 per cent. of fat removed, the powders will show, on analysis, from 25—32 per cent. fat, 6—8.5 per cent. ash, 3—4.5 per cent. fibre, 11—17 per cent. nitrogenous matter, 11—19.5 per cent. starch, and 4—5 per cent. moisture. If the cocoa powders have been rendered “soluble” by treatment with ammonia or ammonium carbonate, or by steam, their analyses will be similar in the main to those of untreated cocoas.

Any additions of foreign matter, other than alkali, must be looked upon as adulteration, and, even if alkali has been used, the addition should be clearly expressed on the wrapper or packet containing the powder.

Cocoa powder is such an excellent medium for conveying materials possessing special medicinal or food values, being a strongly flavoured and palatable article of food, that it is frequently used as the base for preparations sold for dietetic purposes.

If, as has been previously shown, additions of foreign matters, such as starch and sugar, which are cheaper and which, consequently, might be used for purposes of adulteration, are made, it is most necessary that the consumer should realise that he is buying cocoa powder with such additions, however innocuous, or even advantageous, to the food value of the preparation their presence may be.

The addition of starch to a cocoa powder serves the double purpose of increasing the carbohydrate content of the preparation and of rendering the powder more digestible for those who are unable to assimilate the more fatty cocoas. There are, on the market, many of these nourishing cocoa powders which contain selected arrowroot and other starches, with or without the addition of sugar, and which are certainly improved, from the food point of view, by such addition.

Eppe's cocoa contains 40 per cent. cocoa, 16 per cent. West Indian arrowroot, and 44 per cent. sugar, from evidence given in the case of *Gibson v. Leaper*, though Ewell \*

\* E. E. Ewell, *Bull. No. 13, U.S. Dept. of Agriculture.*



gives the following analysis: 3.15 per cent. ash (alkalinity as  $K_2O = 0.6$ ), 25.94 per cent. fat, 1.51 per cent. fibre, 26.0 per cent. sugar, and much arrowroot starch.

Under this heading must certainly be included chocolate powders which, consisting of powdered chocolate, or cocoa and sugar and starch, give similar analyses to that of Epps's cocoa. There is, however, this difference, that the latter, being a standard proprietary article, is mixed with great care and from carefully selected ingredients, whilst some of the chocolate powders that have come under the author's notice have not shown much careful selection of ingredients and have, more frequently than not, been freely mixed with cocoa shell powder. Chocolate powders are, as a rule, very cheap, but there is no reason why they should be nasty, and, if good clean starch has been added to powdered chocolate, the resulting preparation should be as good and wholesome as the best proprietary article.

The chocolate powder prepared by the Chocolat Menier Company showed the following analysis, according to Ewell: 1.40 per cent. ash (alkalinity as  $K_2O = 0.47$ ), 21.31 per cent. fat, 1.10 per cent. fibre, 58 per cent. sugar, and no added starch.

J. Bell, in his "Analysis and Adulteration of Foods," also gives an analysis of a prepared cocoa thus: 4.95 per cent. moisture, 24.94 per cent. fat, 23.03 per cent. sugar, 19.19 per cent. starch, 2.24 per cent. nitrogen; the starch and sugar were both added.

Clayton\* has made analyses of different samples of cocoa essence, which consisted of partially defatted and otherwise treated cacao; a very complete investigation into the component parts of the ash, obtained from them, was made.

Recommendations have been made, from time to time, to

\* E. G. Clayton, *Chem. News*, 1902, lxxxvi., 31.

treat the cocoa powder with boiling sugar, and many patents have been taken out to this end.

Greiser, in an English patent, 1909, suggested that moisture and volatile bodies, that affect the flavour, should be removed in a uniform manner by heating the finely-ground cocoa (with or without the addition of sugar) *in vacuo* at, say, 70° to 90° C. Or, the mass might first be boiled *in vacuo*, then melted with a suitable proportion of sugar and again boiled *in vacuo*.

Langen, in a French patent, 1909, proposed to treat the partially defatted cocoa with a boiling solution of sugar in a vessel fitted with a suitable stirrer. The mixture was stirred until cold, and the mass of chocolate, thus obtained, powdered and pressed into moulds or mixed with milk powder. By this treatment, he claimed to obtain a readily soluble cocoa or chocolate for use as a beverage.

Many more such instances could be cited, though they are only worthy of consideration as showing the extent to which the subject of rendering cocoa "soluble" has been studied. It must be admitted that the majority of the patents taken out are not only of little value for making the cocoa "soluble," but the processes employed usually spoil the aroma of the cocoa and are quite impracticable to work outside the laboratory or experimental room.

Foreign matters with medicinal and other special virtues are frequently added to cocoa powders, amongst others being acorn and barley meal, malt, milk powder, dried egg-albumen, saccharin instead of sugar for persons suffering from diabetes, kola seeds, peptone and other such materials.

In a U.S. patent, 1910, Collett and Eckardt have recommended the addition to cocoa and chocolate of one or more ferments, capable of producing lactic acid from carbohydrates without evolution of gas.

There can, however, be only a very limited sale for all these kinds of preparations, and, as this work deals essentially with cocoa and chocolate and not with all the possible combinations that might be made with other materials, further consideration of them has not been considered necessary.

## CHAPTER XV

### PREPARATION OF CHOCOLATE—MIXING—REFINING

UNSWEETENED chocolate should be prepared exclusively from the roasted kernels of the cacao bean, with or without the addition of a small quantity of flavouring matter. The chocolate should contain at least 45 per cent. of cacao butter, the natural fat of the cacao bean.

Unsweetened chocolate, which is seldom met with in confectioners' shops, but which is usually sold to manufacturers of sweet chocolate who do not possess their own roasting, nibbing and milling plant, is prepared from the cacao mass running from the grinding mill, previously described in Chapter XIII. In fact, the milled, roasted cacao nibs constitute in themselves pure unsweetened chocolate, and, consequently, on analysis, the latter will show figures similar to those of the nibs from which it has been made.

The cacao mass flowing from the mill is in liquid form and, according to the degree of grinding to which it has been subjected, will be found to be in a more or less finely divided state. In order to obtain a smooth homogeneous paste, it is necessary to pass the cacao mass through a series of granite or steel rollers which, running at a high speed, reduce the chocolate to the finest possible form.

The rollers or refiners will be described fully later in the chapter, when the refining of sweetened chocolate is dealt with.

The reduction of cacao nibs to paste may, of course, be brought about in other ways than by passing them through

rollers. A "melangeur" may be used, or the cacao liquor "Disc" machine which is so popular in America. There are, however, good and sufficient reasons why cacao should not be ground in contact with iron or steel, as, even after roasting, the cacao nibs contain an appreciable quantity of moisture which, with the tannin in the cacao, is apt to impart a metallic flavour to the chocolate or cocoa. After the addition of sugar and cacao butter to the nibs, however, the percentage of moisture in the mass is considerably reduced, and this fact, in addition to a further loss of moisture during mixing, makes the effect of iron upon the chocolate mass negligible. This point is considered later, in the chapter dealing with short-cuts to chocolate manufacture.

Sweetened chocolate should contain the kernels of roasted and shelled cacao bean, with the addition of not more than 60 per cent. of sugar, with or without a small quantity of flavouring matter.

The base of sweetened chocolate will, therefore, be pure unsweetened chocolate or the cacao mass running from the mills. To this base is added a sufficiency of sugar and flavouring matter, in such a way that a smooth homogeneous product shall result.

### **Method of Mixing.**

The mixing of the sugar and flavouring matters with the cacao mass is conducted in a large revolving pan in which run a pair of granite rollers ("melangeur"). The pan, revolving horizontally, carries the chocolate mass under the rollers which, being capable of adjustment up and down, are themselves caused to revolve by the friction of the paste passing under them. One or both of the granite rollers may be also power-driven.

It is advisable to give the cacao mass some thirty to forty-five minutes' working in the mixer or "melangeur" previous to introducing the sugar, as, in this way, a certain

homogeneity is obtained in the base, and the work of the refining machines is somewhat facilitated.

The sugar should be ground in a disintegrator or sugar mill and passed through a fine lawn mesh. It is usual to dry the sugar before being mixed in with the cacao mass, but the importance of this point is discussed elsewhere.

Much time will be saved if the "melangeur" is heated up to a temperature  $5^{\circ}$  or  $6^{\circ}$  above the melting-point of the cacao butter, say, to  $40^{\circ}$  C., and, in order to bring about immediate blending of the sugar and cacao mass, both ingredients should be heated to about  $35^{\circ}$  C. before being placed in the "melangeur."

The chief uses of the "melangeur," which has been briefly described, are to obtain the complete blending of the ingredients, to reduce the mass to a finer state of division, and to lighten the paste. The first object is brought about by the revolving pan, travelling at a fairly high speed, carrying the mass under and round the rollers, assisted by two scrapers that turn the paste in the required direction. The constant friction of the pan, which travels at a higher speed than the rollers, rubbing on the cacao mass at the two obstructions, raises the temperature of the whole and gradually reduces the particles to a finer state of division.

After the paste has been passed through the refining rollers, the mass is dry and lifeless, and it is necessary to place it again in the "melangeur," to recover some of its life and limpidity. This is brought about by the constant friction of the chocolate mass on the rollers and by the re-melting and re-distribution of the cacao butter which has dried up in the process of refining.

As will be seen later, better results are obtained by alternate refining on granite and steel rollers, with an intermediate or subsequent treatment in the "melangeur." Since, however, the tendency has been to reduce the number of refinings and to increase the intensity of levigation,

granite rollers have been largely replaced by the steel refiners which, undoubtedly, are very effective in reducing cacao and sugar particles to an extremely fine state of division in the shortest possible time. Saving of time has been further responsible for the introduction of the "Bausman Disc" machine which, with proper handling, is capable of giving good results with chocolate. For those who consider quantity and speed of output before the quality of their product, the complete equipment of a chocolate factory is composed of mixing kettles and "Bausman Disc" machines. There are many such factories in existence, especially in America, though the author can say, without prejudice, that the results are not those of which to be particularly proud.

#### **The "Melangeur."**

The "melangeur," illustrated, is admirably suited for this work. Each roller is fitted with a raising device, which enables an easier starting of the machine after stoppage, does not allow thumping of the rollers on the granite bottom of the pan and causes an easier and more rapid grip on the larger pieces of sugar and cacao, if the latter is put into the pan in lumps.

The "melangeur" is capable of being heated by a steam coil, placed immediately beneath the pan, so that the temperature can be quickly raised to the required point before the charge is introduced. Such a machine occupies a floor space of  $7\frac{1}{2}$  feet by 6 feet and is  $5\frac{1}{2}$  feet high. Iron dust-proof covers can be provided with these "melangeurs" at a little additional cost and will often prove advantageous, when the chocolate paste is run continuously in the machine for any length of time.

"Melangeurs" can be bought with one or two rollers, simple or conical shaped according to requirements, and the capacity of the machine can be made to hold a charge of



FIG. 11.—No. 7 Large, Patented "Melangeur," with Automatic Discharger coupled with Three-roll Upright Refiner. (See p. 204.)

*By permission of Messrs. Joseph Baker, Sons, and Perkins, Ltd.*




anything from  $\frac{1}{2}$  cwt. to 10 cwt. In the machine, illustrated (Fig. 11), the "mélangeur" is shown fitted with an automatic discharger, coupled to a three-roll upright refiner.

The dry sugar, flavouring matters and cacao paste having been well mixed in the "mélangeur," the crude chocolate will be in a semi-liquid form, somewhat resembling a very soft dough. The mass will be slightly gritty to the teeth, due to the particles of sugar and the coarser grains of the milled cacao, and must undergo at least one refining process before anything in the nature of a finished chocolate is obtained.

If the idea of the manufacturer is merely to mix his ingredients together, there is no reason why a steam-jacketed mixer or kneader should not replace the "mélangeur" which, as has been pointed out, has also a refining effect and is particularly adapted for reducing a dried paste to a semi-liquid condition. In America, the steam kettle is commonly used for this purpose, and can be looked upon purely as a mixer. A clever refinement of this principle is shown in a machine made by Messrs. Joseph Baker, Sons, and Perkins, Ltd.—a water-jacketed "mélangeur" which is recommended especially for melting down and for amalgamating added cacao butter or substitutes with the chocolate paste. The "mélangeur" granite runners revolve on a stone bed, and grinding and mixing are thus accomplished whilst the tempering is going on. This machine, with a capacity of 1,500 lbs., occupies about 5 feet 9 inches by 5 feet floor space, is 4 feet 10 inches high, and is well suited for restoring limpidity to a chocolate paste after refining, or for preparing the original mixing, to bring it to a condition fit for refining.

#### **The Refiner.**

 The refiner consists of a series of connected rollers which revolve at different rates of speed, allowing the

chocolate mass to pass between them to be ground both by crushing and by tearing.

An extremely hard form of granite, diorite, has been found to be the most serviceable material with which to construct the rollers, and the quarries, from which the granite of the granite refiners was obtained, were, in 1914, in the hands of the principal makers of chocolate machinery, such as Messrs. Lehmann. Since the war, several other hard granites have been tried with complete success, and the manufacturers of

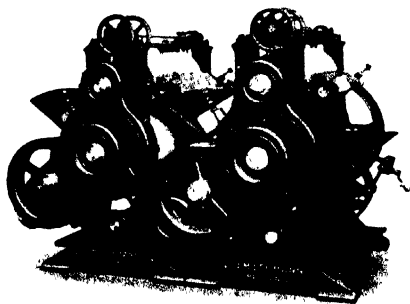


FIG. 12.- Battery of two Granite or Porcelain Three-roll Refiners (See p. 210.)

*By permission of Messrs. Bramwell & Co., London*

chocolate machinery in this country are independent of the German stones. Porcelain rolls have also proved thoroughly satisfactory.

Many modern refiners have cylinders of polished chilled steel, which not only reduce the chocolate to a finer state of division in a given time than can be obtained with a granite set, but they have the additional advantage of being able to be water-cooled—a necessity which arises on the cylinders becoming heated from over-friction or continual running.

The rollers in the older-fashioned granite refiners were placed in a horizontal position, whilst the modern steel or granite roller machines are upright, of which the former has a parallel water-cooling system attached.

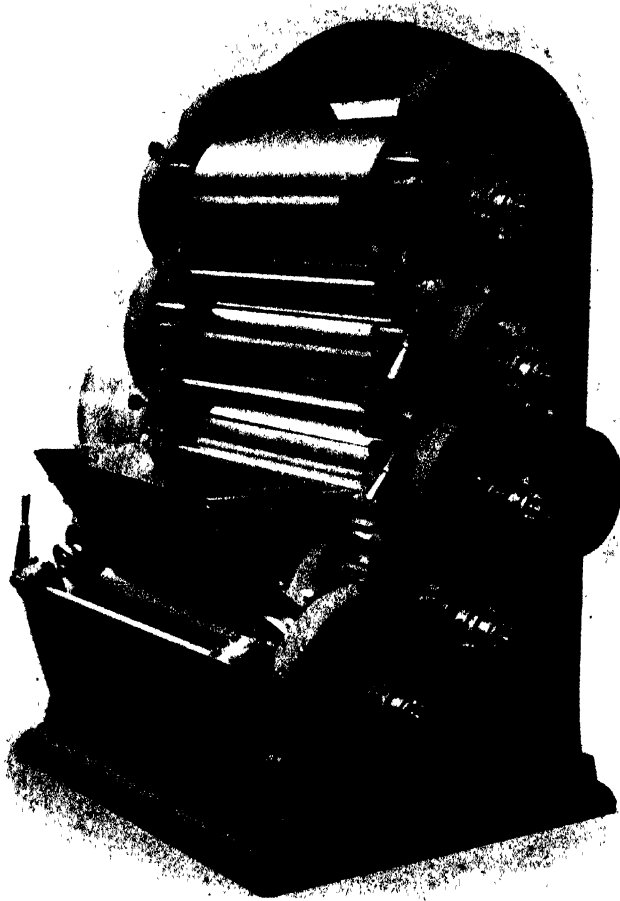


FIG. 13.—Patent Five-roll Upright Refiner, with Chilled Rolls and Spring Bearings. (See p. 208.)

*By permission of Messrs. Joseph Baker, Sons, and Perkins, Ltd.*

Upright steel roller refiners are now made by all makers of chocolate machinery, one of the illustrations showing a machine made by Messrs. Joseph Baker, Sons, and Perkins,



FIG. 14. -Five-roll Steel Refiner, with Chilled Metal Rollers, all Water-cooled. (See p. 209.)

*By permission of Messrs. Bramoak & Co., London*

Ltd., the other by Messrs. Lehmann, of Dresden (Figs. 13 and 14).

The Baker machine has been specially designed to bring

w.c.

14

about a higher speed, larger output and finer grinding than could be obtained on the ordinary granite rollers.

The rollers are of chilled steel, carried on lever arms, with spring-bearings and hand-lever adjustment. These advantages enable a much closer adjustment of the rollers to be made, and, being of chilled metal, they are much harder, will take a finer polish and remain truer in use than if made of granite. The spring bearings and the fact that the cylinders or rollers are water-cooled make it possible to run the machine at high speeds, ensuring a large output and great efficiency.

The five-roll upright refiner with chilled steel rollers and appliances, as described, occupies a space  $4\frac{1}{2}$  feet wide by  $7\frac{3}{4}$  feet long by  $6\frac{3}{4}$  feet high.

If necessary, a battery of refiners can be formed, the chocolate paste from the last roller of the first set being transferred by means of a scraper to the hopper of the next. The advantage of this system is that, where a chocolate needs and can stand a second or third consecutive refining, time is saved by automatically feeding the once-refined chocolate through a second or third set of refiners, without having to wait to transfer it to pass through the same refiner a second or third time.

The chocolate mass is taken from the "mélangeur" straight to the hopper of the refiner in which the first roller is revolving slowly, carrying on its surface sufficient chocolate to transfer it to the next, which travels at a higher speed. The upper sets of rollers revolve still faster and, being set close, grind the chocolate to a fine state. A scraper removes the chocolate from the last roller, either into pans placed beneath or into the hopper of the next set.

All chocolate, for whatever purpose it may be intended, should be passed through the refiners once or twice, the best quality of eating-chocolate being refined as many as six or eight times, between various periods of rest, given to it

in the hot cupboard or hot room, and further treatments in the "melangeur."

It must be understood that, if the grinding in the cacao mill has been satisfactory, the cacao particles will be very small. In any case they are soft, and, if no husk or germ has been allowed to enter the cacao mass, the particles will be readily crushed by a short treatment on a refiner. The sugar particles, on the other hand, though also very small, if the crystals have been crushed in a disintegrator or other suitable mill and the powder subsequently wind-sifted, are hard and appear gritty between the teeth. A very small quantity of sugar, insufficiently ground, will give this effect, and it is not uncommon for a certain amount of the chocolate mass to pass through, untouched, on the outer edges of the refining rollers, if the latter are not absolutely true. This fact is another cogent reason for obtaining machinery from the best engineers manufacturing cocoa and chocolate plant, since the finishing of refining rollers is an art, in which only those who appreciate the importance of perfectly true rollers indulge.

The author, from his earliest connection with chocolate, has always been amazed at the number of refinings to which it seemed necessary to submit chocolate. There is little doubt that, by very careful adjustment of the refining rollers and the use of sugar reduced to an impalpable powder before mixing with the cacao mass, very fine chocolate can be made with only one refining. There is, however, this objection, that chocolate, so refined, must be restored, after refining, with a large amount of cacao butter, in order to bring it to a state sufficiently limpid for moulding. It was for this reason that the author investigated the whole question and, starting on the assumption that it was sugar that required the close refining, ascertained the size of particles that could be felt as grit between the teeth. The investigation led to a number of interesting observations

and, finally, to the able research work of Bradley,\* who, in conjunction with Messrs. Peek, Frean and Company, Ltd., Rowntree and Company, Ltd., and Joseph Baker and Sons, Ltd., elaborated a system for the preparation of sugar which could be as easily crushed as the cacao particles. The system of manufacture of so-called "amorphous" sugar is described later and opens up the only possibility of employing a short-cut to chocolate manufacture, known to the author, without deterioration of the product.

The chocolate mass, after the first mixing, whichever sugar has been used, is in a soft, doughy state, but, after being passed through the refiner once, it is reduced to a dry, light powder, similar in appearance to cocoa powder, only more flaky.

This is partly the result of cooling down on the rollers, which should never be run whilst hot, and partly from the crushing of the sugar particles, which tends to dry the chocolate by exposing a greater superficial area to be covered by the fat.

In this dry, powdery state, the chocolate is not in a fit condition to undergo further immediate refining, but it should be placed in a pan in the hot cupboard, to recover, and then run for a short time in the "mélangeur," before being again passed through the refiner. The hot cupboard is especially useful in factories with a continuous call upon their kettles or "mélangeurs."

By constant consecutive refining, there is a tendency to "fatigue" the chocolate, that is to say, to reduce it to a powdery state from which it will not recover without addition of more cacao butter. This may often be the case after the first refining, if too large a quantity of sugar has been used or if the cacao nibs are not of a very fatty nature, but it may be avoided in later refinings by giving the cacao mass a rest in the hot cupboard, when the butter will tend

\* Brit. Pat. Spec., 132,200, 1919.

to come to the surface and will be found once again to permeate the whole mass, which can be readily reduced to

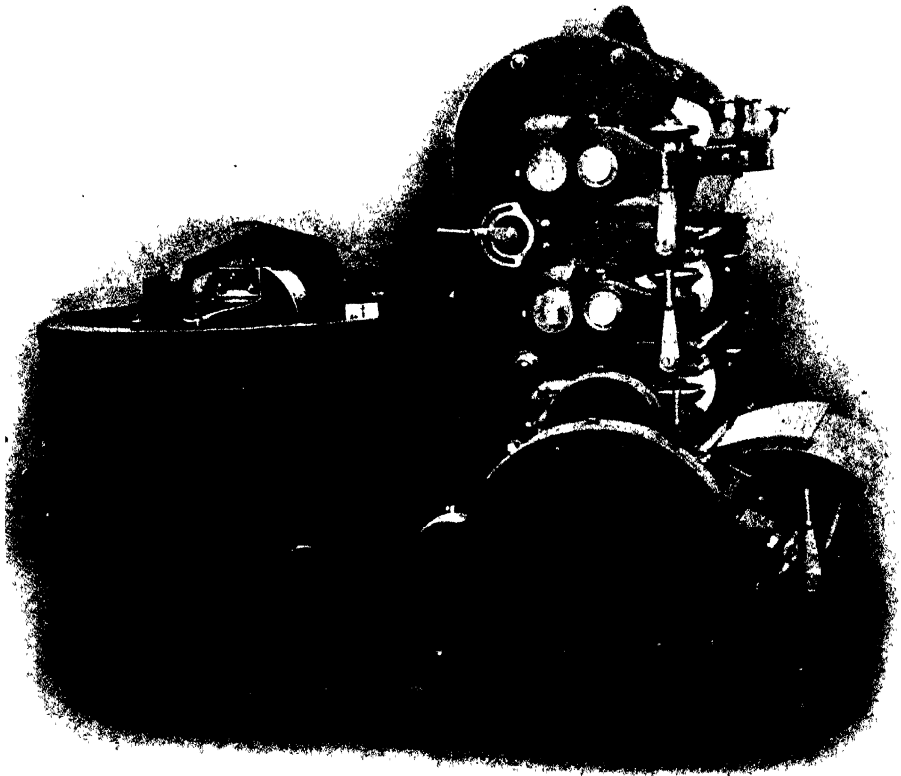


FIG. 15.—Combined Refiner and Water-jacketed "Melangeur." (See p. 213)

*By permission of Messrs. Joseph Baker, Sons, and Perkins, Ltd.*

liquid state, as soon as it has run for a short time in a "melangeur." A combined refiner and water-jacketed "melangeur" is illustrated (Fig. 15).



There is little doubt that chocolate "fatigues" more easily on granite than on water-cooled steel roller refiners, and especially so is this the case when milk chocolates, containing a great deal of body matters, are undergoing the process.

The alternate refining on granite and steel refiners has certain advantages which are discussed fully in a later place. It is doubtful, however, whether granite rollers will ever be employed to any great extent in chocolate factories again, since the output of the product from the steel refiners is very much greater than, and the quality but little inferior to, that from the granite refiners.

The degree to which chocolate is refined is entirely a matter of taste and requirements; that for use for covering sweets or fancy centres and biscuits requires less refining than the finest eating-chocolate, though there are many people who still prefer the coarse and gritty chocolate bars for eating purposes to the smooth and finely-milled product, now so common on the market. This subject has been discussed in all its aspects in the next chapter.

Speed of production does not result in the highest standard of quality, and it is undoubtedly true that the treatment in the "melangeur," for an extended period, enhances the flavour and consistency of the finished product and, in time, would reduce the chocolate to as fine a state of division as would be attained by the use of a refiner. The same result may be obtained in the "conche" machine which is described in detail later. This prolonged treatment, however, must be an expensive item in a factory where the machinery is driven by steam or electricity, though, in a water-driven factory, the cost is comparatively insignificant. In the former case, therefore, greater reliance must be placed on the efficiency of the refiner which reduces the chocolate to a fine state in a short space of time, the

"*melangeur*" being used for obtaining the required consistency.

It must not be imagined that refining consists of merely running the chocolate paste through the refiners, for several important points must be considered before the required result and output can be obtained. The judicious adjustment of the rollers is a point of no small importance, whilst the speed of running, in a great many chocolate factories, is often too rapid. Both these points must be carefully watched, and the difficulties can be overcome by placing a skilled and competent man in charge of the refiners. The result of running the rollers at too high a speed will be the heating and drying of the paste, which, as has already been shown, can only be cured by addition of more cacao butter or by prolonged treatment in the hot cupboard or the "*melangeur*," both remedies resulting in loss of profit, in ingredients, time, and labour. It is a mistaken idea of economy to purchase cheap refiners or those made by unknown makers. Much time and experiment has been put into the development of the modern refiner by the best engineers, and the chocolate manufacturer would do well to take advantage of the experience acquired by such experts as Messrs. Joseph Baker, Sons, and Perkins, Ltd., and Messrs. Lehmann.

#### **The Hot Cupboard or Closet.**

The hot cupboard, or hot room, which has frequently been mentioned, is one of the most essential fittings of the chocolate factory, especially if the "*melangeurs*" are working up to their full capacity.

It has already been shown that chocolate pastes, after being "*fatigued*" by the refiners, recover their texture after being placed in the hot cupboards or closets for a few hours. Many pastes, that seem too dry ever to recover, will be found to revive, if left in the hot cupboard for twenty-four hours, and no extra addition of cacao butter will be needed.

The hot cupboard is also employed for melting cacao butter and chocolate base and for warming the sugar, prior to mixing them in the "mélangeur."

The heating chamber consists of an iron frame with iron shelves and is fitted with overlapping double swing doors. The heating is effected by steam in the space at the bottom of the chamber. The usual storage capacity is about 600 lbs. of chocolate to each yard of space, and the complete unit measures about 10 feet long by  $2\frac{3}{4}$  feet deep by  $6\frac{1}{2}$  feet high. For extension purposes, additional lengths of 3 to 4 feet can be obtained.

The hot room is, of course, even more suitable for dealing with large quantities of chocolate than is the hot cupboard, and the temperature to be maintained should be as high as possible, consistent with the materials to be handled. The milk-fat in milk chocolate is liable to turn rancid after long exposures to very high temperatures, so that intelligence must control the temperature and time of treatment in the hot room, as in every other operation during the manufacture of chocolate. The correct temperatures for working milk chocolate are discussed in Chapter XXI.

## CHAPTER XVI

### SHORT-CUTS TO THE MANUFACTURE OF CHOCOLATE— INFLUENCE OF METHOD ON QUALITY

ELSEWHERE, it has been observed that people eat chocolate because they like it and not because it is good for them. In the light of recent years, this observation must be modified, seeing that there is a huge increase in the consumption of sugar *per* head which has at least some physiological significance. Without doubt, the very great demand by the public, at the present time, for sugar or sweet-stuffs has arisen, in Europe at any rate, from the shortage of sugar during the years of war. In America, when the statistics are published, the huge increased consumption of sugar *per* head will astonish many and may be mainly attributed to prohibition of alcohol and to the greatly increased wages among the workers.

Both the pleasure and profit derived by eating sweet-stuffs, whether as cakes, puddings or candies, having been denied or strictly curtailed to the masses in Europe, there is a natural reaction in favour of consumption of these good things at the present time, whilst the absence of alcoholic liquor in America has also created an additional demand for sweet things in that country.

From 1889 up to 1914, the consumption of sugar *per* head had only increased 22·5 per cent. in the United Kingdom, whilst America showed an increase of 70·6 per cent., and Germany 323·4 per cent. Yet, in 1914—15, the actual consumption *per* head in the three countries, respectively, was 89·69 lbs., 86·04 lbs., and 74·95 lbs. In the following table are shown the figures for these countries from

1910—11 to 1914—15, so that modern pre-war statistics may be considered :—

	U.K. lbs.	U.S.A. lbs.	Germany. lbs.
1910—11 . .	91·64 .	77·15 .	47·89
1911—12 . .	85·38 .	82·43 .	41·27
1912—13 . .	95·52 .	85·04 .	49·26
1913—14 . .	93·37 .	89·14 .	45·13
1914—15 . .	89·69 .	86·04 .	74·95

With the cessation of war, we may expect an increase in America, owing to the greater production and less restriction in consumption of sugar, to prohibition and, finally, to the great wealth of the country and to the prosperity of its working classes.

A slight increase in the United Kingdom may be expected, and a decrease in Germany, owing to her inflated consumption in 1914—15, due to the fact that she could not export her surplus of sugar and that the present sugar production is lower than in pre-war days.\*

How far the consumption *per* head in America will be modified in the first year or two after the war, owing to the exporting of larger quantities of sugar to Germany and to Europe generally, it is not possible to say. She will, also, doubtless figure as a very large exporter of chocolates and candies to the war-ravaged countries.

It may be truly said that, both in Europe and America, the end of the war saw, immediately, a very greatly increased demand for sweet-stuffs, and among them chocolate. So great was the demand that quality shrank into the background, and it is doubtful whether, for many years to come, the public will recover its discriminating taste, or, if it does, whether the manufacturer will pander to it. Particularly does this apply to America, where, at the moment, there is no bottom to the market, and where, rightly or

\* A large quantity of sugar was used in Germany, during the war, for the manufacture of glycerine, a process which was developed to a high state of efficiency.

wrongly, special attention is directed to mass-production, to cope with the demands. So far as America is concerned, this state of affairs may continue for a long time, and, even with the greatly enlarged businesses and the erection of numberless new chocolate houses, the rapidly increasing population and its powers of expenditure will prevent competition among the manufacturers from being felt to any great extent. The limitation of sugar production seems to be the only factor that may adversely affect the output of chocolate in the United States, and the writer is not one of those who believes that, simultaneously with the cheapening of sugar at some future date, the demand for chocolate will fall off greatly in that country.

The question of quality is not an easy one to discuss. In the year 1920 in which this is written, quality is of less account than it was in early 1914, that is to say, chocolate will sell itself, to a very large extent, in any part of the world at the present time, provided that it is palatably sweet and that it tastes of chocolate. There is also a very large sale of *so-called* chocolate, even without these reservations, which could not have found a market in pre-war days. The question of quality is, therefore, of less significance now than it should be. Yet, owing to the fact that old conditions must obtain sooner or later, quality must be again considered, if future development of the trade is to take place.\*

America and Europe are not on the same plane with regard to quality, the requirements of the general public in either continent being as different as chalk from cheese—a more apt simile than appears on the surface.

In America, the cult of the smooth chocolate has not been developed, and, to the European, the majority of

\* It is interesting to note that, since these lines were written, even the best manufacturers of chocolate have compromised on this point and are providing both good high-priced chocolate and cheap low-grade stuff, of which the latter is similar to the worst war chocolate and could not have found a market in pre-war days.

American milk and plain chocolates taste crude and unfinished. To the American, the European chocolates appear slimy and insipid, and the writer has heard milk chocolate from Europe frequently described in the United States as "Cowdy." In justice to America, it should be remarked that judgment should not be passed upon the American chocolates recently flooding the English markets. Much harm has been done to the American chocolate trade through exporting to Europe products of very poor quality, probably on the assumption that anything would sell during the shortage. As a statement of fact, there are many manufacturers in the United States capable of producing as good a chocolate as any to be found on this side of the water, but, unfortunately, too few show their capability.

This is certainly not the place to discuss the *pros* and *cons*, advantages and disadvantages, superiority and inferiority, of either product. Every country has some strong partiality to specific qualities or strength of flavour, a partiality derived from the pleasing or displeasing effect of aromas upon the sense of smell. Thus, the prevailing tastes for vanilla in France and for lemon in England are examples of one form of partiality. It is no question of good or bad taste, but only one of like or dislike.

In chocolates, plain and milk, there enters also the question of the feel on the tongue, roughness or smoothness, and, by gradual stages, Europe has transferred its affection from caramels, their smoothness and flavour, to milk chocolate, thence to the smooth "fondant" chocolate, now so universally popular. In this way, the coarse chocolate of old has largely disappeared.

America does not seem to be passing through the same stages, and it possesses very few houses which specialise in either fine, smooth milk or "fondant" chocolates. Coarse varieties of milk chocolate are most common, and the very great popularity of "fancy chocolates" with filled centres,

as distinct from straight tablet chocolate, also accounts to some degree for lack of attention paid to the chocolate itself.

It is most common to find, in the United States, houses, with a huge output of covered chocolates or fancy chocolates, that do not make even their own covering-chocolate, but buy from other factories which specialise in this direction. In Europe, it is only the very small and, usually, retail chocolate-makers that buy the covering-chocolate in this way.

In speaking of American chocolate, therefore, as crude, it is not intended to decry that product—far from it—for it has the virtues of popularity and great sales in that continent. Yet it is intended to convey the suggestion that there is less attention paid to its manufacture than to the production of most of the European chocolate. Firstly, owing to the huge quantities produced by the factories struggling to cope with the demands, and, secondly, from the fact that the chocolate can be sold as fast as made, the manufacturer is, clearly, less able to concern himself about, and less interested in maintaining, a high standard than if he were up against competition.

The remark made by the writer in the United States, when discussing this same thing, that “chocolate made even on the family mangle would sell well,” is not far from the truth, the degree of refining and the art of manufacture of a very large number of the chocolates on the American market requiring very little more machinery than one or two of these pieces of household furniture. Generally speaking, the manufacturers are aware of this fact, and some have resorted to devices but little removed from a mangle.

It is, clearly, a question of public taste, and it does not seem even desirable to attempt to substitute much finer chocolate, produced at a greater cost, when huge quantities of that more readily prepared can be sold with the utmost ease. On the other hand, every manufacturer in every part of the



world must ask himself, at some time or the other, whether the throwing together of the necessary ingredients, with a rough mixing and a certain amount of grinding or refining, gives the best results as seen in the finished article.

By such a process, chocolate, and good chocolate, can be made, but only if the ingredients are of the best, and provided certain fundamental principles are observed. But individuality cannot be obtained by these means, and considerable care in detail must be expended if the best flavoured chocolates are to result. It is only so that Cadbury's "Mexican" and "Bournville" chocolates, Terry's "Bitter" chocolate, Chocolat Lindt, Peek Frean's "Meltis," Marquis vanilla chocolate, and many other of the familiar brands with individuality have won their deserved popularity in the face of keenest competition. It is true that, as soon as competition ceased during the war—for it was then rather a question of supplying an unprecedented demand with acute shortage of all the necessary ingredients—the quality of these well-known makes fell off. It could not have been otherwise, but now it rests with the manufacturer to determine how soon the public will recover its old pre-war discrimination and to return to his good standards.

This general argument has brought us to a point which is of the greatest importance to the future of the chocolate industry, namely, as to whether there is any short-cut to the making of chocolate. It is hardly necessary to say that such a question is not being considered here for the first time. It is of the utmost interest to the manufacturer to know whether all the costly machinery, at present found in an up-to-date factory, is really necessary, seeing that chocolate, in the old days, was made in a pestle and mortar and that, in the evolution of the art of chocolate manufacture, new machines have been constantly added and old machines improved until, in order to produce the finest "fondant" or milk chocolates, complex machines, of highest credit to the engineer, have

to-day become apparent necessities. The answer is simple to understand, yet somewhat complex to put in black and white.

Firstly, come in the matter of competition, met by the manufacturer, and the necessity of finding a chocolate of sufficient individuality and attractiveness to tickle the popular fancy. In order to secure character in a chocolate, a specific flavour must be either added or else secured, by a special treatment of the chocolate during manufacture. In the first case, additional machinery is not required, but the devices of the manufacturer may extend to any of the machines adapted for cooling or heating or special grinding, or to the invention of a new machine, such as the "horizontal" or "conche," to secure a required result.

With regard to the "conche," there has never been a machine so much discussed. What is the use of a "conche"? What result, unobtainable on other machines, does it effect, etc.? The "conche" provides a machine specially adapted for running continuously for a large number of hours with the minimum amount of friction in the bearings and mechanical parts of the machine, but with a maximum amount of friction and movement given to the chocolate, which can be kept at any required temperature. A "mélangeur" might do the same work, but it is both a clumsy device for this purpose and is open to the objections that there is greater friction involved in the machinery parts and that it is less easily adapted for temperature control. Added to this, the "conche" keeps every portion of the chocolate paste in continuous and even violent motion, which secures a result unobtainable in a "mélangeur," chaser or kettle. But the "conche" might be just as well excluded from a factory, or substituted by a kettle, if the chocolate is not allowed to remain under the agitating and rubbing movement for at least thirty-six hours. In the "conche," too, an after-development and improvement of flavour can be

encouraged by heat, provided, of course, allowance has been made in the roast, in the simplest and most economical way and without loss of aroma that might, and naturally does, occur in an open "melangeur" or kettle.

The next consideration for the manufacturer is as to how much the finest qualities of chocolate owe their virtues to the machinery employed. At the outset, it may be asserted that you cannot make good chocolate out of bad materials and that good materials may be spoilt by inattention to detail, even with the best machinery, but more easily by the use of bad machinery. Taking the first of these sub-considerations, even an efficient roaster may provide inconsistent roasts, or burn the beans, if care is not exercised. A badly adjusted husking machine may allow too much husk to find its way into the chocolate mass; a cocoa mill, with stones set too close, may burn the cacao mass, and so on. Through every stage of manufacture, attention to detail and to adjustment of machines is essential to the production of good chocolate.

As to the use of bad machines, it is clear that machinery incapable of careful adjustment is open to all the dangers of good machinery without the advantage of cure, secured by attention. Again, machinery, made by engineers not fully cognisant of the materials that have to be handled on the machine, is liable to all sorts of imperfections, to the detriment of the chocolate. Of this class of machinery, the writer speaks feelingly and can but warn prospective chocolate manufacturers against them.

Next in importance, is the old debatable point of the respective merits of steel over granite rollers in the process of refining. When steel refiners were first introduced, they revolutionised to a great extent the chocolate industry. They enabled chocolate makers to refine more closely and quickly their chocolate products, but, even to this day, there are many manufacturers and technical men who are not

entirely converted to their use. When steel rolls were put on the market, it was stated by almost all the chocolate manufacturers that the chocolate would taste metallic, and many maintain, to-day, that it does so. The writer is not, and was not, one of these, though he is aware that it is possible to make chocolate taste metallic by passing it through steel refiners, if the ordinary precautions in practice are not carried out.

To-day, so accustomed to the idea of steel refiners have manufacturers become, that steel kettles, steel-bedded "conches," and even steel cocoa mills and chocolate mills have become the order of the day. The scarcity of the best granite has had something to do with the great change of front, and, generally speaking, the product has not suffered by the change.

How far can this adoption of steel and iron, instead of granite and stone, be carried? Formerly, it was universally held, and still is in some of the best factories, that the less the chocolate comes into contact with iron the better. The writer is strongly of this opinion, for the reason that cacao contains tannins which, in the presence of moisture, quickly act upon iron, producing iron tannates of dark colour and strong metallic taste. Previous to refining, the chocolate paste should have been sufficiently heated, in the processes of roasting and mixing in the heated "mélangeur," to render it free from moisture, for all practical purposes. Therefore, the action of refining on steel rollers is negligible so far as the attack upon iron is concerned. Any process including and following refining is safely conducted in iron vessels for this reason, provided that access of moisture is prevented and that the chocolate is not allowed to absorb water from the air during the time elapsing between the processes. But, up to the point of refining, the cacao mass itself retains often as much as 5 per cent. of water, which, in the writer's opinion, backed by experience, is far too high to allow

contact with iron to be made safely and without the cacao mass acquiring a metallic flavour.

Steel mills for cacao mass, or for direct grinding of the mass with sugar, are, to the writer's way of thinking, inadmissible in the preparation of the best chocolate. Further than this, the action of a cocoa mill is not only to reduce the nibs to the finest state of division possible within the limits of the time and space, but, also, to rupture the cell sacs and to free the enclosed fat : in other words, it is desirable to rend or tear the fibre rather than to grind. The best method of doing this is between two stones.

With regard to refining, much the same remarks apply, especially in the latter sense. The action of rapidly revolving steel rollers is to crush, and, indeed, they crush and refine very efficiently, since there is less need to rend the fibres of the cacao once that has been done on the stones of the cocoa mill. But, those who have tried refining from the cold chocolate block on a granite mill will admit that that method, too, has its advantages, which, in the production of quality, far outweigh to a chosen few the advantages of speed and mechanical efficiency of later types of refiners.

This argument for superiority of granite over steel is often spoken of as "hair-splitting," and, in these days of large outputs, most manufacturers have been forced to admit that the differences in the flavour of the chocolate, resulting from the two procedures, are of so little importance that it is doubtful whether the public would realise them, whilst the efficiency and speed of grinding by the modern steel refiners are so great that he cannot afford to ignore the financial advantage to be derived.

Each one to his taste, his conscience and his pocket !

A great many attempts have been made to reduce the quantity of machinery and the time taken for refining chocolate. Some of these efforts have met with commercial success, but, except in the last few years, claims have not been

made that the finest chocolate can be produced by these short-cut processes. It should be stated, in defence of the manufacturers of these machines, that the claims, at the outset, have been moderate, but that it is the chocolate maker who has often seen in the adoption of these machines a cheap method of manufacture of his product which he passes off on the public as finest eating-chocolate. So long as the public can be induced to buy such preparations by clever advertisement, or so long as it lacks discrimination, the use of short-cut machines will be found eminently satisfactory to the chocolate manufacturer. Our own opinion is that the best eating-chocolate cannot be produced on these machines, for reasons stated earlier in the chapter, whilst we readily acknowledge the value of such inventions as the "Bausman Disc" machine, the "Walker" machine and other similar devices for the manufacture of covering-chocolates in a cheap and simple manner.

It is not our wish, therefore, to decry these efforts at improvement in chocolate manufacture, just because certain extravagant claims are made. On the contrary, we wish to point out the direction in which the chocolate manufacturer may, with advantage, adopt such inventions. For the manufacture of covering-chocolate, for coating fancy chocolates, biscuits, etc., there is no doubt that the "Bausman Disc" machine, especially, will be found eminently serviceable and profitable. Indeed, in America, the author was struck with the great possibilities of being able to equip a chocolate factory with kettles and "Bausman Disc" machines only. It was not, however, until the author looked into the product that he realised that, with even the greatest care expended, the resulting material lacked the finish that we expect to find in good eating-chocolates. In America, as we have observed before, the public is not particularly discriminating, and, for this reason alone, the "Bausman Disc" machine finds, at the present time, considerable

popularity among manufacturers whose business is simple in that they can sell all the chocolate they can make without serious consideration having to be given to quality. On the other hand, there are many excellent firms who would scorn to sell to their public covering-chocolate as a fine eating-chocolate.

### 1. The "Bausman Disc" Machine.

The "Bausman Disc" reducer is used for the same purpose as the cocoa mills, *i.e.*, to reduce the nibs to a liquor in as fine a state of division as possible. The principle underlying this machine, as in the "Bausman Disc" refiner also, is the passage of the nibs between three grooved, steel discs, of which the centre one alone revolves. The more modern machines are water-cooled, so that the heat of friction shall not affect the cacao mass detrimentally, and, in this arrangement, lies the great advantage over stone mills, which, at best, are bad conductors of heat, owing to the material of which they are made. It is certain that the faces of the outside discs can be kept cool, even when close grinding is in operation, though the author admits to the dislike of treating cacao mass on steel rollers or discs, or of allowing it to come into contact with iron or steel of any sort.

The quantity of cacao liquor that can be reduced on a "Bausman Disc" machine varies from 200 to 300 lbs. *per* hour, according to the closeness of the grinding, a triple mill being capable of an output of 150 to 250 lbs. *per* hour, though this has been increased somewhat of late, owing to improved methods of cutting the stones. There is, therefore, a saving both in output and space by adopting the use of a "Disc" machine, since the floor space occupied by the latter is only 2 feet 6 inches by 4 feet 4 inches. The power required for the "Disc" reducer is only 5 h.p., the speed of the driving pulley being 350 to 400 r.p.m. The horse-power

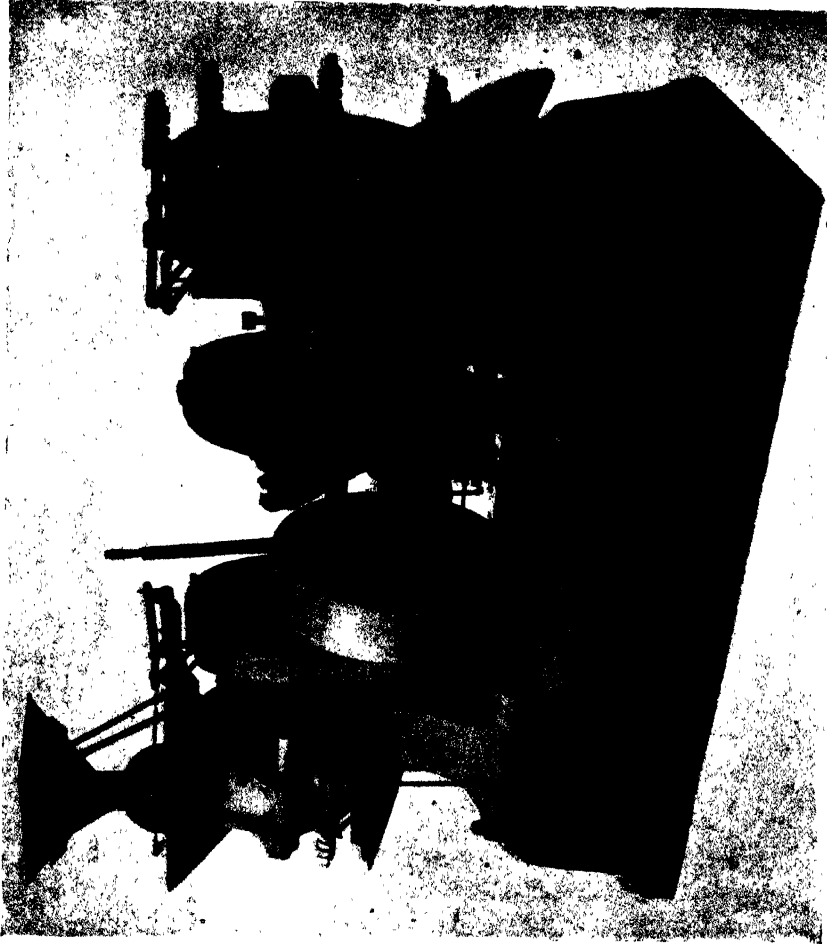


FIG. 16.—"Bausman Disc" Reducer and Refiner. (See p. 228.)  
*By permission of Messrs. S.A.T. Jeunissen & Co. Par.*

required to drive a Triple Mill is about the same, though the speed at which the stones revolve is considerably slower.

The "Bausman Disc" refiner is run on much the same principles as the reducer, though the discs are larger in



diameter. The power necessary to drive the refiner is 15 h.p., the number of revolutions, required, being 300 to 350 *per* minute. The refiner is especially suitable for reducing chocolate paste, after mixing, to a suitable smoothness for use as covering-chocolate, but, as has already been indicated, some manufacturers use the machine for preparing eating-chocolate which is not of the smoothest variety. The dies require re-cutting fairly frequently, owing to the large output, 200 to 300 lbs. per hour according to the degree of fineness required, and the more modern machines are equipped with easily moved discs, which, it is recommended, should be replaced when showing signs of wear. The chief disadvantage of this refiner lies in the necessity of adding large quantities of cacao butter in order to secure a satisfactory output.

It is claimed by the makers that the "Bausman Disc" machines are very sanitary, in that the cacao liquor and chocolate paste are protected from dust and external contamination. Further, it is claimed that the aroma is conserved by keeping the preparation away from the air. An illustration of the "Bausman Disc" machine (to left), and the "Bausman Disc" refiner (to right) is shown in Fig. 16.

## 2. The "Walker" Machine.

The "Walker" machine consists of two conical, grooved cylinders, the one situated above the other, each fitting closely with, and revolving within, a hollow, grooved conical cylinder. The chocolate paste passes from one to the other and undergoes a crushing or disintegration in its journey. It is necessary, in order to obtain the best results, to crush the nibs to a liquor before mixing with sugar and passing through the cylinders. The sugar can be quite coarse and crystalline, and only a medium covering-chocolate can be made on this machine.

The author understood, when he was in America, that a considerable improvement had been made by the inventor and that excellent results had been obtained with the new model. No machine was seen, however, during the author's stay in the United States.

### 3. The "**Bramigk**" Ball Machine.

There have been several types of this machine constructed, all based on the tumbler principle in which heavy steel or stone balls are carried round on the inner side of a cylinder or drum and made to fall upon the material to be crushed or ground. The constant friction also levigates the mass, and the machine has the advantage of being able to treat the nibs and sugar direct, without preliminary grinding, though it possesses the serious disadvantage of being extremely noisy, being dubbed by the workmen in one factory, visited by the author, the "Hell-on-Earth" machine.

### 4. "**Gloor**" Patent.

English patent, 1915, 7,041, and French patent, 1915, 479,067, relate a process for the manufacture of all sorts of eating- and dessert chocolates and covering-chocolates by simultaneously cooking, roasting and pulverising the cacao beans and caramelising the sugar. The patent has laudable and ambitious objects—to reduce the cost of manufacture of such chocolates and, through this rapid roasting and cooking process, to retain all the cacao butter present in the beans and all the valuable chemical constituents, required in standard special chocolates, and thereby to obtain a much finer flavoured chocolate than hitherto. The temperature necessary for the cooking, etc., up to as high as 350° F., is produced by friction instead of by steam or fire, and the whole operation is complete in thirty-five to sixty minutes. For beans containing a high percentage of fat, 250° F. is recommended as the maximum temperature. The less

cacao butter, present in the beans, the higher the temperature may be allowed to rise.

Any suitable machine may be used, the only essential being, according to the inventor, that sufficient churning and rubbing of the particles should take place, to liberate the necessary heat. "For example, as a type of apparatus that will perform the process, a machine can be used wherein two sets of rapidly rotating paddles or the like, interlocking in their paths in a well-known manner, are used to effect the purpose." Needless to say, the speed must be high. The chocolates produced by this process, as seen by the author, do not call for great enthusiasm as to flavour, though the state of division is very fine.

#### **5. Combined Refiner and Water-jacketed "Melangeur" (Fig. 15), Water-jacketed "Melangeur," or Continuous Refining Machine**

may each be used to produce satisfactory covering-chocolate and do not call for special mention. Messrs. Joseph Baker, Sons, and Perkins, Ltd., make each of these combinations and single machines, and can provide further information as to their output, cost, etc.

#### **6. "Amorphous" Sugar.**

Instead of looking to the modern chocolate machines, which must be recognised as fulfilling their duties very efficiently, the author, for many years, has been inclined to look to the modification of the ingredients, in order to effect increase of output without deterioration of the product. Any attempt to reduce appreciably the time taken in, or to cut out any of the operations of, chocolate manufacture has, so far, been met by failure, in that the product, resulting, has been inferior in quality and aroma. There is a great deal to be said for time-honoured customs where the quality of a manufactured article is concerned, especially when dealing

with materials that possess elusive qualities of aroma and consistency.

In the early days, it mattered little how much time was spent in preparing a product so long as the article, required, was eventually obtained. To-day, perhaps, the public is less exacting, yet its attitude does not seem to justify the adoption, by the manufacturer, of methods that clearly are efficient only in increasing output. Modern inclination has been towards a very smooth chocolate, usually termed "fondant" chocolate, which requires long treatment on the refiner and (or) in a "conche" machine, and the addition of a large quantity of cacao butter. The method of making "fondant" chocolate is discussed in a following chapter, but it seems a suitable place here to consider an especially advantageous system for the preparation of the most important ingredient in chocolate manufacture.

Hard crystal sugar is usually employed in making chocolate, and, when it is realised that it is the grittiness of this sugar that it is required to refine away or to round off, the former being accomplished on the refiner, the latter in a heated "conche" machine, the importance attached to this ingredient by the author will not be found to be misplaced. After numerous experiments, both on the small and large scale, the preparation of "amorphous" sugar has now come to be recognised as a distinct step in the right direction for the manufacture of chocolate. The principle underlying the idea is simple. Firstly, a soft sugar is prepared instead of a hard one, and, secondly, after appreciating the fact that it is possible to over-refine, by reducing the particles beyond a certain degree of fineness when the grittiness felt by the teeth is long passed, the sugar, so produced, is of such a nature that, on one crushing in the refiner, it will be at once reduced to the size when it can no longer be felt as grit between the teeth. Over-refining is also extravagant in cacao butter, in the smaller the particles are reduced the larger is the

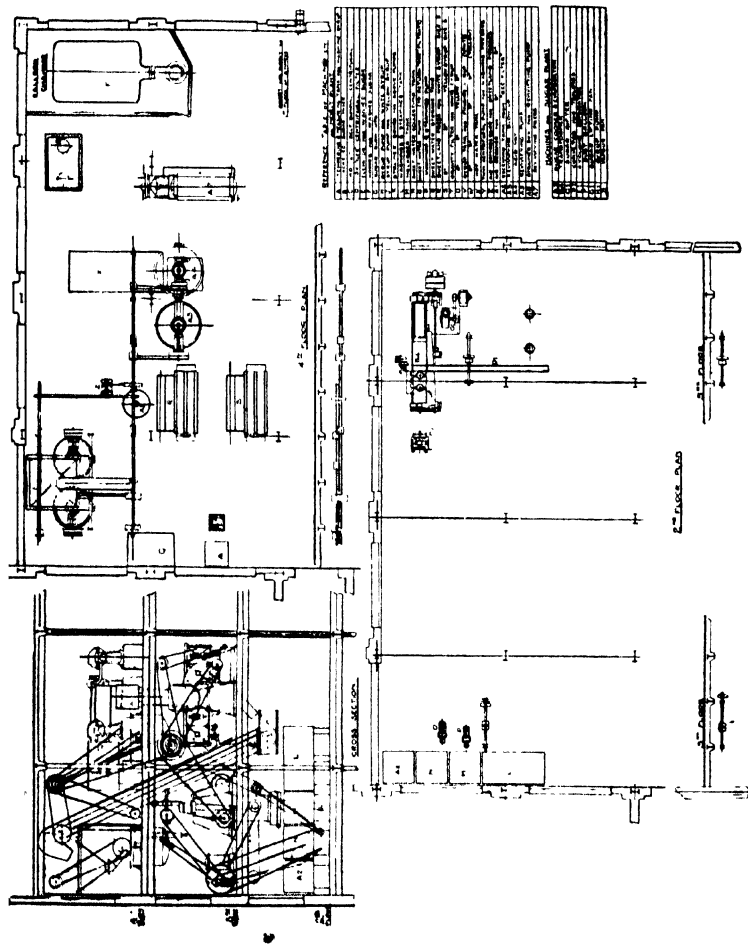


FIG. 17.—Cross-section and Second- and Fourth- Floor Plans of "Amorphous" Sugar Plant, with Refinery System. (See p. 232.)

By permission of Messrs. Joseph Baker, Sons, and Perkins, Ltd.

superficial area to be covered and the drier the resulting paste, or the greater the quantity of cacao butter necessary

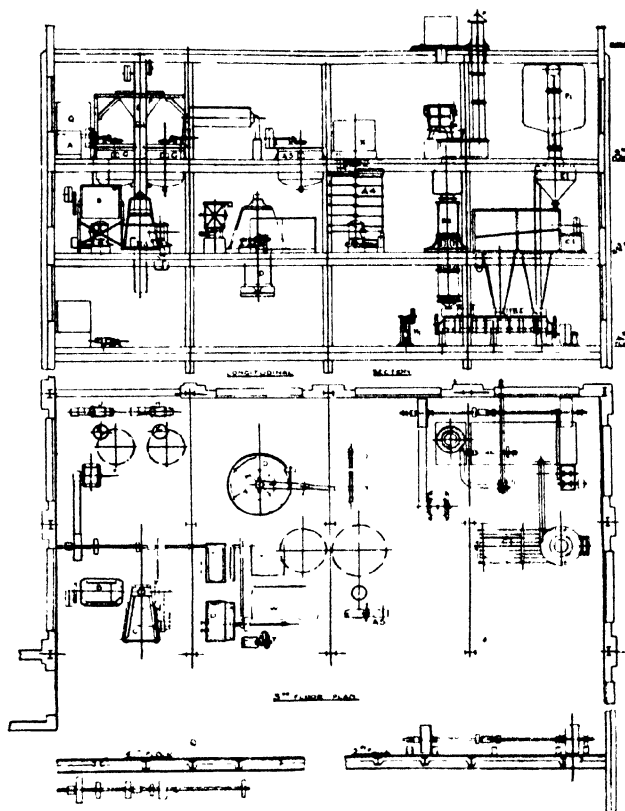


FIG. 18.—Elevation and Third-Floor Plan of "Amorphous" Sugar Plant, with Refinery System. (See p. 232.)

*By permission of Messrs. Joseph Baker, Sons, and Perkins, Ltd.*

to be added, to bring the chocolate to its correct consistency. Many "fondant" chocolates on the market suffer from this

defect, since their analyses show unnecessarily high percentages of fat.

Considerable improvements have been made in this process by Bradley, Carr, Messrs. Peck, Frean & Co., Ltd., Messrs. Rowntree & Co., Ltd., and Messrs. Joseph Baker & Sons, Ltd., and the method of manufacture of the sugar has been described in English patent 132,200, 1919. This patent describes a refinement of the processes, mentioned in English patents 28,296, 28,297 of 1903 and 4,112 of 1904, in which the manufacture of "amorphous" sugar is given in full detail. It should be remarked, incidentally, that a very great additional advantage to the chocolate manufacturer, especially if he makes confectionery also, is to be found in the fact that the system may be adapted to include a miniature refinery, in which white syrups or sugars can be produced from raw sugars, a matter of very considerable importance in these times, besides providing handsome remuneration. A line drawing of the system is shown in Figs. 17 and 18, and the results, obtained in chocolate by the use of "amorphous" sugar, are to be seen in the photomicrographs shown in Chapter XXVIII., Figs. 35 to 38.

## CHAPTER XVII

### MOULDING CHOCOLATE TABLETS, CROQUETTES, NEAPOLITANS AND FANCY SHAPES; TAPPING, COOLING, WRAPPING

THE chocolate, after the necessary smoothness and consistency have been obtained by treatment in the "melangeur" and refiners, is placed in the hot cupboards or hot room till required for moulding. Each tin in the cupboard should be clearly marked to indicate the grade of the chocolate and date of manufacture.

It is desirable to knead the chocolate paste prior to moulding, as the temperature in the hot cupboard, of  $50^{\circ}$  to  $60^{\circ}$  C., may have caused separation of the solids from the fat which would rise to the surface.

The chocolate-kneading machine, sometimes called a "chaser," is usually a small high-speed one-roller "melangeur" which may require heating, according to the temperature maintained in the factory and the coldness of the day.

Steam-jacketed stirring kettles are more commonly employed for keeping the chocolate mass at the correct temperature, and, by keeping the stirrers in motion, the chocolate is maintained in a suitable homogeneous condition ready for the moulding operation.

When the chocolate paste has acquired the correct consistency, which does not usually take longer than the time necessary to mix it thoroughly, it is taken from the kneading machine and placed on a wooden or marble table and spread in shallow rows. The reason for this operation is to bring down the temperature of the chocolate paste to one reasonably low enough for passing through an air-



extracting machine, automatic weigher, or other desirable labour-saving device, immediately prior to moulding. When kettles are used, this process is not necessary, as the chocolate can be maintained in them at any required temperature.

#### **Air-Extracting Machine.**

Where an air-extracting machine is employed, the chocolate, at a temperature of 32° to 34° C., must be fed into the machine, warmed to a temperature similar to that of the paste.

The chocolate, in passing from the hopper, is forced by a helical axle into a small receptacle, whence it escapes through a conical orifice and is cut by a simple device into required lengths or weights.

In a machine designed by Messrs. Joseph Baker, Sons, and Perkins, Ltd., an apparatus, somewhat resembling the cartridge cylinder of a revolver, is placed in front of the outlet of the air-extracting machine and is fitted with six tubes, in each of which works a piston. Each tube is presented, in turn, in front of the outlet and, as soon as filled, is automatically emptied by the piston so as to deliver, if required, on to moulds placed on a continuous travelling band. By altering the size of the tubes, the machine can be adjusted to weigh from  $\frac{1}{4}$  lb. to  $\frac{1}{2}$  lb., or from 1 oz. up to  $\frac{1}{4}$  lb.

The overall dimensions of the machine are 5 $\frac{1}{4}$  feet long by 2 feet wide by 4 feet high.

The air-extracting machines work very well for medium and soft pastes, but irregular and rough lengths will emerge from the outlet if the chocolate is too dry.

In many factories, the air-extractor is dispensed with, especially where hard dry pastes are employed, and in such cases, the chocolate is pressed directly into the clean dry moulds, when the subsequent action of the tapping table brings the air-bubbles to the top where they burst.

**Automatic Tempering Machine.**

As has already been stated, before moulding, it is necessary to get the mass of chocolate to a certain and constant temperature, if the best results are to be secured. The same remark applies to covering-chocolate before it is placed upon the centres, whether by hand or by machine. To this end,

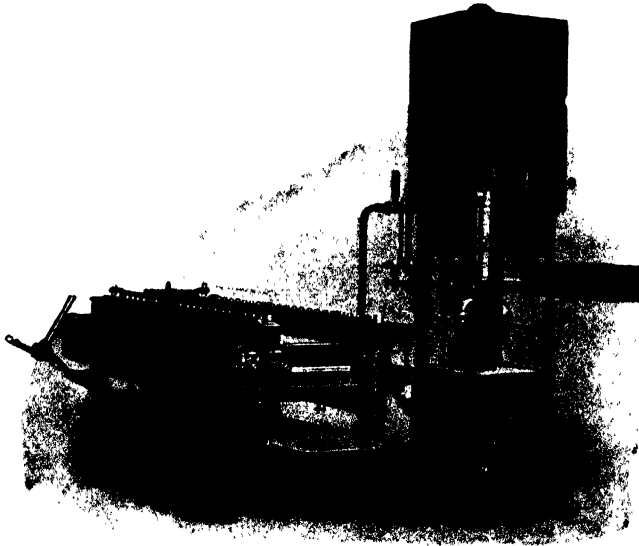


FIG. 19 --Automatic Tempering Machine for Stiff and Liquid Chocolate, Milk Chocolate, Coverings, etc. (See p. 240.)

*By permission of Messrs. Bramley & Co., London.*

“melangeurs” mixers, steam kettles, etc., may be used. Such appliances are, however, slow and laborious in operation. and, since it is a well recognised fact that the taste, appearance and break of the finished chocolate depend, to a very great extent, upon the temperature of the material during the moulding process, it is not surprising that the engineers

of the chocolate industry have devoted much time and attention to an automatic device for achieving the same end.

The Automatic Tempering Machine, illustrated (Fig. 19), is probably the latest word in machines for bringing chocolate automatically and continuously to any required temperature for covering or moulding. In one minute from the time the chocolate is fed into the hopper, the mass is evenly and consistently heated to the temperature required. The machine is simple in operation, and two special devices, of a very ingenious design, automatically maintain the temperature, once they have been adjusted.

The machine consists of a cast iron stand, about 9 feet long, supporting a number of hollow rollers which are arranged in pairs. There are two water-tanks, and a rotary pump raises the water from the lower to the upper tank where the water is heated by means of live steam, thence, by a series of copper tubes, to the hollow rollers. The pipes, connecting the rollers with the hot and cold water supplies, are so arranged that different temperatures can be obtained on the various rollers—that is to say, one section of the rollers can be made warmer or colder than the rest, at will.

The chocolate is taken from the store room and, without any previous preparation, is fed into the hopper. It then passes horizontally between the two sets of revolving rollers and is delivered, in about one minute, to the other end of the machine. While passing over the rollers, it takes up the required temperature and, at the end of the machine, is ready for moulding or for covering fancy centres. Thermometers are provided, whenever necessary, for observing the temperature of the chocolate. The horse-power for driving the machine is small, from  $1\frac{1}{2}$  h.p. to  $2\frac{1}{2}$  h.p. for machines for tempering  $1\frac{1}{2}$  to 3 tons *per* 10-hour day, and  $2\frac{1}{2}$  to 5 tons *per* 10-hour day, respectively.

### Moulding.

Assuming that the chocolate is ready to be moulded, whether or no it has passed through the air-extractor and weighing machines, and is at a temperature of  $32^{\circ}$  C., the paste of dry chocolate may be pressed by hand into the moulds, or, if of the softer varieties, spread with a palette knife.

The moulds should be of polished white metal, thoroughly clean and dry, warmed to the same temperature as the paste. These precautions are most necessary, as the finish of the tablet or moulded article depends entirely upon the care with which the moulds are treated. Dull spots on the surface of the finished, moulded chocolate are the results of dull, dirty moulds, or they may be caused by the moulds being cooler than the paste pressed into them. The gray top surface "break" of the chocolate may also be put down to this same cause.

The correct temperature of chocolate paste for moulding is from  $32^{\circ}$  C. to  $34^{\circ}$  C., though many figures have been published to show the varying temperatures for moulding at different times of the year. Such variations of moulding temperatures should not occur in factories kept at a reasonably constant temperature throughout the year, say  $20^{\circ}$  C.—a matter of no great difficulty in these days of efficient heating and cooling systems. Small tablets, however, should not be moulded below  $32^{\circ}$  C., whilst larger masses of chocolate will hold the heat they contain the longer and may, consequently, be moulded as low as, say,  $30^{\circ}$  C.

If variations of temperature do occur in the atmosphere of the moulding room, it is obviously necessary to increase the temperature of moulding, during the cold, and to lower it, during the hot weather. Experience is the best guide to the correct temperature for moulding during fluctuations of atmospheric temperature, though  $5^{\circ}$  C. on each side of the limits, mentioned, will certainly meet all requirements.

It is advisable to make use of a thermometer to regulate

the moulding temperature, though, here again, experience steps in, and it will be found that persons well versed in moulding chocolate, and especially those who are constantly handling the same variety, can tell, as accurately as any thermometer, when the correct temperature has been reached, by the feel of the consistency of the paste or by the heat of the chocolate felt on the lip.

If the chocolate is to be sold by weight and to be moulded, the weighed paste is pressed evenly over the mould; if merely in tablets without a guaranteed weight, sufficient paste to fill the mould is pressed or filled in with a palette-knife, and the surplus chocolate removed by running the knife over the surface of the mould.

Many curious defects occur on moulded chocolate tablets, such as white streaks and dull patches. Most manufacturers pay no attention to these slight imperfections, which are liable to appear on tablets in the most carefully controlled factories. The author has investigated many of these faults and has collected the following information which may prove serviceable to the chocolate manufacturer who takes a pride in his product. For "fondant" chocolate, the best temperature at which to place the paste into the moulds is between 32° and 34° C. The moulds must be perfectly cleaned, free from moisture, fat and finger marks. Dented or broken moulds also produce unsightly tablets and should be discarded.

The temperature of the "setting" room into which the moulds are placed, or through which they pass, should not fall below 1° C., nor rise above 8° C. in normal climates with a normal room temperature of about 17° to 18° C. If the lower temperature is employed, the transition from the "setting" room to the packing room should be slow, the temperature gradually rising to that of the normal room. This will prevent the formation on the tablets of dew which would cause other troubles, such as sweating and discolora-

tion, if the tablets were packed damp. White streaks in the chocolate are often caused by dry chocolate, left on the palette-knives, finding its way into the chocolate paste. The palette-knives being too cold may also cause the same defect.

If the chocolate is cooled too slowly, or if the chocolate is placed in the moulds at too high or too low a temperature, it will turn white and dull.

Chocolate, allowed to cool at ordinary room temperature, never sets hard, but always remains soft and doughy. Much loss of "snap" is due to this cause. Dull patches appear on the moulded face of the chocolate which look like dew, but which will not evaporate or char on heating.

Under the microscope, all cases of whiteness and streakiness appear to be due to the same cause, that is, to circular patches of crystals, which are almost certainly of cacao butter, appearing on or just under the surface of the chocolate.

A few actual experiments will illustrate the effect of rapidity and temperature of cooling on the appearance of moulded chocolate :—

(1) Room temperature,  $23^{\circ}$  C. ; wet bulb of hygrometer,  $19^{\circ}$  C.

A "plaque" of filled moulds was placed in front of a fan and cooled down in about two hours. The tablets were all white and streaky.

(2) Chocolate paste, at  $37^{\circ}$  C., was taken, moulded and placed in the cold room at  $5^{\circ}$  C. When set, which it only did with the greatest difficulty, the chocolate returned to a granular, soft and rather doughy state on standing at ordinary room temperature of  $23^{\circ}$  C. Considerable discoloration was also noticed.

(3) Chocolate at  $34^{\circ}$  C. was cooled in an open room about  $20^{\circ}$  C. The tablets were white, streaky and mottled inside, and dull patches, which looked like, but were not, dew,

showed both on the moulds and the moulded faces of the chocolate tablets.

(4) Chocolate at 30° C. was moulded and cooled at about 4° C. The result was slight streakiness on the moulded faces of the tablets.

The defects, enumerated above, arising in moulded tablets of chocolate may also be caused by too hot or too cold, dirty, damp or greasy moulds. Care expended in cleaning and tempering moulds is well repaid by the excellence of the finish of the tablets.

When moulding milk chocolate, the paste may be taken straight from the "conche," if at the correct temperature, or after tempering to about 34° C., which has been found to be the best moulding temperature. Cooling is conducted at about 7° C. for from 30 to 35 minutes, and it should be remembered that milk chocolate cools much more slowly than plain chocolate.

#### **Moulds.**

The appearance of a finished chocolate depends upon the temperature at which the moulding is conducted, the nature of the ingredients and, especially, upon the quality of the moulds. The temperature of the chocolate and of the moulds is most important in securing a good finish to the surface of chocolate, but no amount of care, expended in this direction, can ensure a high glaze if the moulds are imperfect. During the tapping or shaking process, the soft chocolate is shaken into intimate contact with every part of the surface of the mould, so that, if there are any faulty apertures, the chocolate, on cooling, hardens into them, and the subsequent knocking-out has to be excessive, reducing considerably the life of the moulds and making the surface of the chocolate tablets unsightly. Undulations and irregularities of the mould, which might easily escape detection on examination, show up very markedly on the chocolate tablet produced in an imperfect mould.

The most satisfactory moulds, of which the writer knows, are those made by Anton Reiche, who are the sole makers of "Plattinol," a special compound metal that takes a very fine polish and that, unlike tin-plate, is not a surfaced metal. The gloss obtained on the chocolate is superb, and the moulds are more solidly constructed and lasting in use than those made of tin. The cost of "Plattinol" moulds is approxi-

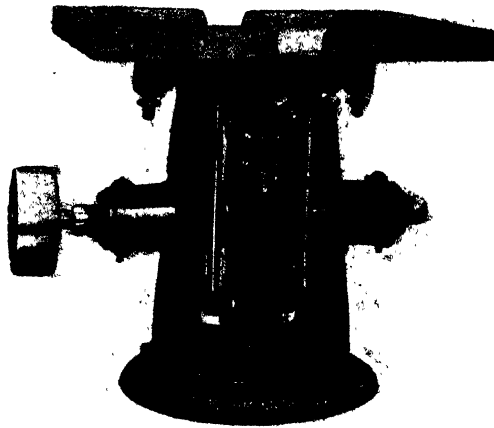


FIG. 20.—Noiseless Shaking Table. (See p. 245.)

*By permission of Messrs. Bramigk & Co., London.*

mately 30 per cent. higher than tinned moulds (at the present time), but the excellence of the result and the longer life make the higher price of no account.

The agents for Anton Reiche in Great Britain and the colonies are Messrs. Bramigk & Co., 92, Fenchurch Street, London, E.C. 3.

#### **The Tapping Table or Shaker.**

The chocolate-filled moulds are then placed on the tapping table (Fig. 20), an apparatus which, by means of a



roughly toothed wheel placed beneath the table-surface, is given a rapid and violent vibrating movement, and consequently, the moulds, placed thereon, suffer the same treatment. The effect of this is to cause the chocolate to run into every corner of the mould, completely filling it, and, by coming into close contact with the polished metal, the chocolate is given a high polish when cold. Any bubbles which may be retained in the paste are brought to the top and burst, but they may be removed by a knife, or by breaking them with the tip of the finger, if the vibration is insufficient to cause them to burst of their own accord.

The noise, created by the bumping of the table-top and of the moulds upon it, is very considerable, and, where many tapping tables are at work, it becomes deafening. There are now, fortunately, on the market noiseless tapping tables which reduce the greater part of this inconvenience with the exception of the noise caused by the moulds themselves. The clattering of the moulds cannot be obviated, as it is necessary to jar them continuously and violently, a result which could not be obtained if a felt or other silencer were placed upon the table.

The tables are adjusted so as to administer anything from a very heavy shaking down to the merest vibration, so that all sizes of moulds, containing the thickest to the thinnest tablets of chocolate, from hard, dry paste to the most liquid, can be efficiently shaken into the moulds and freed from bubbles.

The average dimensions of a tapping table are 3 feet long, 2 feet wide, and 3 feet high, but batteries of tapping tables are now made, which greatly facilitate the work.

Batteries of any required length can be obtained from Messrs. Joseph Baker, Sons and Perkins, Ltd., or from Messrs. Bramigk & Co., both of whom have also specialised in continuous mould-filling, tapping and cooling plants, described later. The moulds are carried over the tapping

tables by cross-bars connected to chains, and the tables can be covered in, if desired. Each table can be separately regulated, so that the exact amount of tapping, necessary to settle the chocolate into the moulds and to remove the air bubbles, can be secured. As the tables are perfectly level and the tapping is evenly distributed, the chocolate paste settles into the moulds evenly also, with the result that tablets always of the same thickness are assured. The importance of this to the manufacturer who uses automatic wrapping machines, and to those interested in the perfection of their product, will at once be recognised. The speed, too, at which the moulds travel over the tables can be regulated by a set of change wheels, supplied with the tables, so that all classes of chocolate pastes can be satisfactorily dealt with.

All the larger tablets of chocolates, such as bars, croquettes, neapolitans, etc., can be moulded in the method described, the smaller chocolate drops, shells, and other small fancy shapes requiring a special system of moulding, to increase the rate of production.

The moulds for the larger sizes of chocolate tablets are made of heavy, specially tinned and polished metal, or of white metal and other alloys, and contain from two to twenty-four impressions, according to the size of the tablet required, whilst the chocolate-drop moulds, or designs of similar size, are stamped out in light tin of much larger dimensions than the heavier moulds, containing as many as twelve dozen impressions.

#### **Drop Press Machine.**

Small mouldings, and even neapolitans and croquettes, are, with advantage, made on the drop press, which mainly consists of a square vessel into which fits a plunger, worked by a screw with a turning wheel attached. The bottom of the vessel is fitted with a removable perforated bottom, the position of the holes corresponding

to the impressions of the mould which is slipped in underneath. After the chocolate, warmed to the correct temperature, has been placed into the receptacle, the plunger is lowered by the turning wheel, and, when gentle pressure is felt on the chocolate mass, a small quantity of the paste is forced through the perforations of the bottom into the corresponding impressions of the mould.

The surplus chocolate is removed from the mould-plate by a scraper, and the chocolate-filled moulds are then placed on the tapping table. In this way, a very large number of moulds can be filled in a short time and at a much greater speed than by hand-filling.

By so arranging the drop press, the chocolate can be made to deposit, on plain sheets of tin, in drops of any size and can be caused to assume a symmetrical, thin and flattened shape by placing the sheet on the tapping table.

The drops can be covered, if desired, with coloured sugar or "hundreds and thousands" before being placed in the cooling chamber. The modern power-driven "Drop Press" is a great improvement on the hand-machine. Particulars of the latest press, made by Messrs. Lehmann, can be obtained from Messrs. Brannig & Co.

#### **Chocolate Coolers.**

When the moulds have been filled and tapped, they should be cooled down, so as to harden the chocolate for knocking-out. It has been shown by experience that the texture and appearance of moulded chocolate are improved by rapid cooling—facts which are probably due to the formation of small crystals of cacao butter when rapidly cooled, whereas slow cooling would produce larger and coarser crystalline formation. On the other hand, there are greater dangers in too-rapid cooling, if the utmost care is not taken to prevent the condensation of moisture, which will occur if the cold tablets are taken into a warmer and moister room

immediately from the cooling chamber. The deposition of moisture may also take place in the cold room itself, if the air is not kept dry by keeping the chamber closed as much as possible.

It is true that the majority of firms make use of artificially cooled rooms or chests in which the temperature is not allowed to rise above zero, mainly for the purpose of accelerating the cooling process, but better, safer and more consistent results will be obtained if a room, kept constantly at a low temperature, of say  $8^{\circ}\text{C}$ ., is employed for cooling down the chocolate. In winter months, this should not be difficult to obtain, but, in the warmer summer days, artificial cooling would have to be resorted to, unless the room were situated well below the level of the ground.

There are many systems for producing low temperatures artificially, and they are all based upon the principle of absorption of heat when a volatile liquid, such as an easily compressible gas in condensed form, is allowed to expand to the gaseous state. The cold, so created, may be allowed to act directly upon the cooling chamber, or to be imparted to solutions of salts, such as of calcium chloride, which, flowing through pipes in and around the chamber, quickly lower the temperature. Liquid ammonia, carbonic acid, and sulphurous acid are chiefly used for producing the cold, and, by a system of alternate expansion and compression, the volatile liquids are reduced first to the gaseous state and then back to the liquid form, the result of the first process being allowed to react in the neighbourhood of the cooling chamber, or upon the brine solution, without loss or escape of the gases.

For large cooling chambers, the brine and ammonia system is most economical. The brine may either circulate in pipes around the chamber, or be in turn deprived of its chill by a strong draught of air blowing through it, as it falls over the cold ammonia pipes.

. In smaller factories, where the cooling requirements are not so great, the carbonic or sulphurous acid system, coupled with circulating brine, will be found to be the most serviceable and efficient and to occupy only a small space.

The drying of the cold air acts as a safeguard against condensation of moisture on the surface of the chocolate, though the same danger by removing the cold, full moulds to a warmer, moister room will still be incurred. To obviate this, it is desirable to bring the temperature of the moulds slowly up to that of the factory, either by passing them on a slow travelling band through chambers of steadily increasing temperature, or by placing them in a room at a temperature between that of the cold room and that of the factory for a short time prior to knocking the chocolate from the moulds.

When the chocolate and moulds have attained the normal temperature of the factory, the moulds should be slightly bent backwards or gently tapped to remove the contents, which may then be packed in tin-foil and wrapper, ready for the market.

The moulds should never be struck hard to remove the chocolate, and, if moulded at the correct temperature, there will be no need, for the tablets will fall out without tapping.

If too high a temperature has been employed during moulding, the chocolate will stick tight to the mould, and the surface will be spotted with dull patches ; if moulded too cold, the surface will be whitish and marbled.

During cooling, the chocolate contracts and generates frictional electricity, which has been attributed to the rubbing of the particles of chocolate on the metallic surface of the mould containing it. A certain Mr. Sanders, a chocolate maker, observed this and found that, by continually heating and cooling the chocolate, the power of generation of electricity gradually disappeared, but that it could be restored by addition of a small quantity of olive oil.

### **Continuous Moulding, Tapping, and Cooling Installation.**

Seeing that moulding operations take so much labour, it is not remarkable that engineering ingenuity has been directed towards the development of a continuous automatic plant, to increase the output and to reduce the wage bill. With the exception of temperature control, all the processes of moulding and tapping are mechanical, and the development of efficient machinery to carry out the work is all that is required.

In applying a mechanical system to a technical process, sufficient latitude must be allowed to enable the machine to handle, with equal efficiency, any variation in the materials. In the development of the chocolate moulding machines this has been the greatest difficulty. It is easy enough to design a machine that will handle either hard, doughy pastes or soft, liquid pastes individually, but to construct a machine that will correctly weigh, to a grain or two, either of these pastes, at the will of the operator, is much more difficult to accomplish.

One machine of this kind, manufactured by Messrs. Savy, Jeanjean et Cie., of Paris, has been described in Fritsch's book "*Fabrication du Chocolat*," 1910, and others by Messrs. Joseph Baker, Sons and Perkins, Ltd., and Messrs. Lehmann, were in existence at that time. In recent years, however, a very great improvement has been made in the continuous automatic mould-filling plant, and, with high price of labour and with the greatly increased output from these modern machines, no large chocolate factory can well afford to be without at least one of these economical systems.

The author has seen the system of Messrs. Joseph Baker, Sons and Perkins, Ltd., at work, and, from his own experience can speak of the excellence of the arrangement and of the finish of the goods obtained.

The mould-filling machine (Fig. 21) consists of a water-jacketed hopper, for maintaining the chocolate at the correct temperature for moulding, and is furnished with mixing

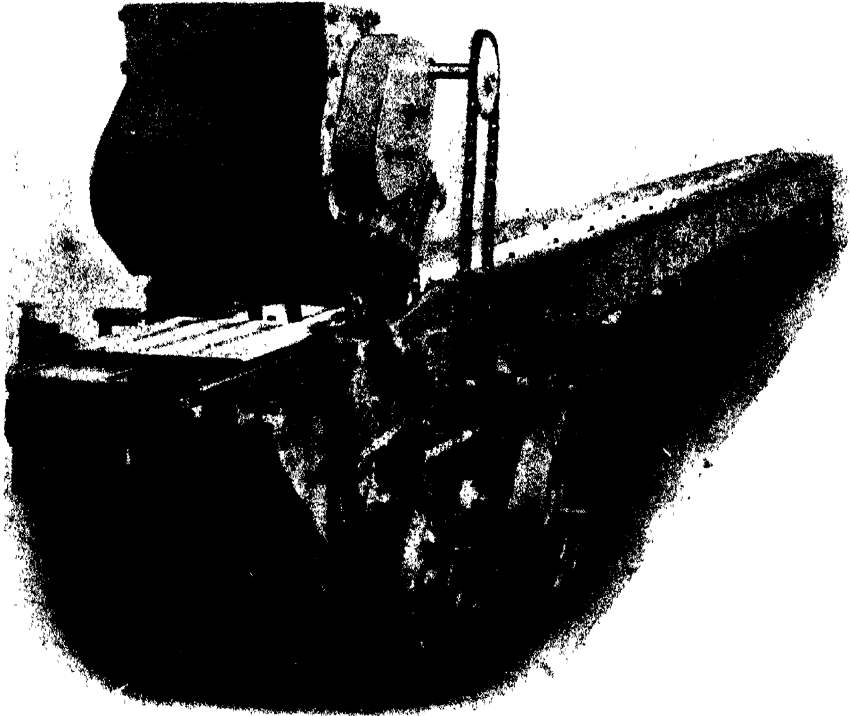


FIG. 21.—Patent Mould-filling Machine, combined with Improved Continuous Tapping Table. (See p. 252.)

*By permission of Messrs. Joseph Baker, Sons and Perkins, Ltd.*

blades which keep the chocolate in motion and of even consistency. A pair of adjustable rollers, fixed to the bottom of the hopper, feed the chocolate into the measuring cylinders which are so constructed as to be able to fill any mould with the exact amount of chocolate paste, soft or hard, that may

be required. There is a cut-off arrangement which leaves no "tails," and the machine is adjustable for different weights and can be regulated whilst running.

According to the consistency of the paste, the machines can be run at varying speeds; with stiff chocolate, some fifteen strokes to the minute, and with lighter pastes, some twenty-five strokes *per* minute will be found suitable. This means that from 60 to 100 moulds *per* minute can be filled, according to the size of the tablets and to the nature of the chocolate.

The machine is made to take a tray of moulds about  $17\frac{1}{2}$  inches wide, outside measurement, and, when working on large tablets ( $\frac{1}{2}$  lb.), will deposit some nine tons of chocolate *per* day. Any size tablets, neapolitans, croquettes, etc., can be deposited by this machine, and, of course, the output will vary according to the size of tablet to be moulded, but the standard machine is made for a maximum deposit of 2 to  $2\frac{1}{4}$  lbs. of chocolate *per* stroke. One operator to feed the machine is all that is required, the remainder of the operation being automatic. The moulds, after passing beneath the depositor, are conveyed on to the tapping tables, over the whole series of which they pass, whilst the chocolate is settled down rapidly and evenly into the moulds. The tapping tables can be adjusted by means of a hand-wheel to fit any required degree of tapping at any point, and the vibration, caused, is more rapid than on the ordinary tables, as this motion has been found to be more effective. For liquid chocolate, seven, for average work, nine, and for very stiff chocolate, twelve tables in series are recommended. A further improvement is to provide the tables with dust-proof covers with heavy plate-glass tops which, besides improving the appearance of the moulding room and safeguarding the chocolate against dust, greatly minimise the noise of the tapping tables.

An operator empties the moulds from the trays on to the



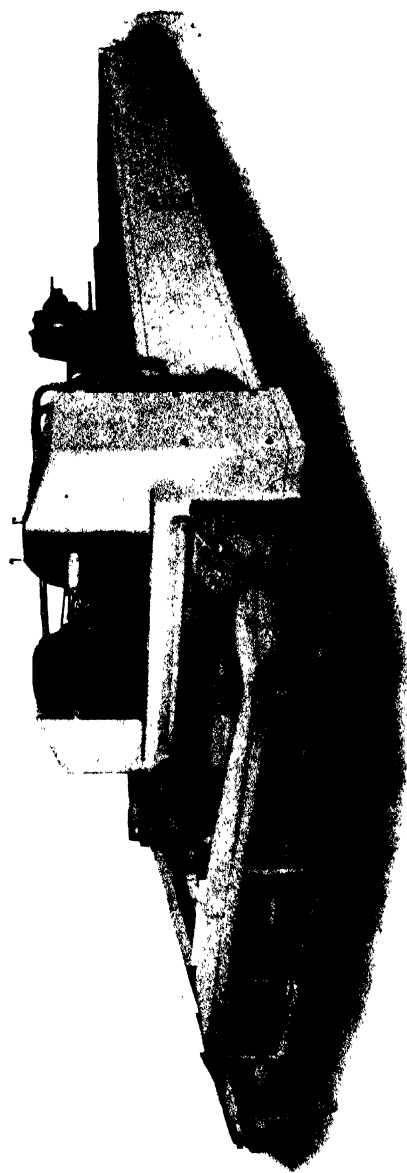


FIG. 22.—Chocolate Moulding, Tapping and Cooling Installation. (See p. 255.)  
*By permission of Messrs. Joseph Baker, Sons and Perkins, Ltd.*

travelling cooler band, whilst the trays are placed on a conveyer band which takes them back to the operator at the moulding machine. The cooler is an insulated wooden chamber, maintained at a low temperature by air, drawn over brine or direct expansion coils, the principle of counter currents by which the coldest air meets the coolest chocolate being usually employed. Thermometers are fitted at the entrance and exit of the cooling chamber, so that the temperature can be observed and controlled. Drains carry away the condensed moisture.

A further refinement of the system is the attachment at the end of the cooler (Fig. 22), where the moulds are transferred to a cross-transfer web which, in turn, delivers them to the de-moulding travelling band running parallel to the cooler. The moulds are taken from the de-moulding web by girls who knock out the chocolate tablets, placing the chocolate on one web that runs to the packing room and the emptied moulds on to another web immediately above the de-moulding band, running back to the moulding machine. The empty moulds pass through a heating system on their return journey and are ready for refilling as soon as they arrive at their destination, which is a table quite close to the moulding machine at the same spot to which the wooden trays are returned from the end of the tapping tables. In this way, labour, space and refrigeration costs are greatly reduced, and the absolute accuracy and uniformity of the working of the machine enable an immense saving to be made.

#### **Chocolate-Weighing and Moulding Machine.**

(Messrs. Lehmann, Dresden.)

The machine, illustrated (Fig. 23), is made by Messrs. Lehmann and has been designed to deal with chocolate pastes of the soft and liquid variety, as well as those of stiff chocolate and nut chocolate. Chocolate cakes, from about  $1\frac{1}{4}$  ozs. to 9 ozs. weight, can be deposited by this machine, which is constructed to deliver, automatically, the paste into each

mould in the form of a ribbon, without soiling the rims of the moulds. From 10,000 to 15,000 chocolate tablets *per* ten

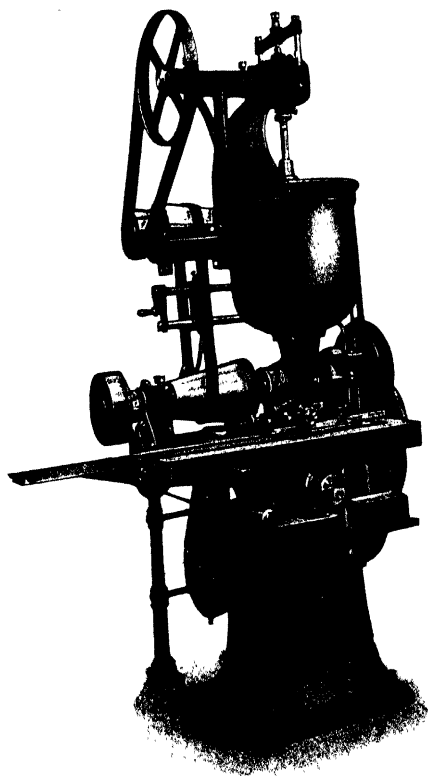


FIG. 23.—Weighing and Moulding Machine, for all classes of Chocolate. (See p. 255.)

*By permission of Messrs. Bramick & Co., London.*

hours can be deposited by this machine, which can be made to deliver the moulds to an endless belt, thence to the tapping tables and cooling chamber.

The machine consists of a hopper, jacketed and fitted with the necessary pipes and connections for steam-heating and water-cooling, so that the correct moulding temperature can easily be maintained. The hopper is also fitted with stirrers and thermometers. The chocolate is discharged through an outlet at the bottom of the hopper, and several nozzles, for moulds of various widths, are supplied with the machine. If the machine is to be used for nut or almond chocolate containing whole nuts, a special stirrer and nozzle are necessary; otherwise no alteration to the machine is required.

### Wrapping.

The purpose of wrapping is twofold: to protect the chocolate from atmospheric moisture and oxidation and from sunlight, and to make an attractive package.

So far as the first is concerned, chocolate deteriorates rapidly on exposure to damp and light, the worst possible conditions being those obtaining in a shop-window lighted by gas, especially in winter time. The first effect of moisture is to dull the surface of the chocolate and, later, to cause small drops of moisture to appear. The drops of water are absorbed by the finely divided sugar which is dissolved, and, even if the water is subsequently evaporated off, the appearance of the chocolate remains unsightly and has all the appearance of stale stock. Sunlight and warmth have the effect, first, of melting the surface of the chocolate and, later, after the chocolate has set again, of turning the surface white, dull and streaky, due to the partial separation of fat and sugar, both light-coloured materials, from the darker cacao matter. How far oxidation is responsible for the deterioration of chocolate in sunlight and warmth it is not easy to say, yet it has been shown that chocolate wrapped in "silver paper" or foil, which is practically air-tight, is less liable to these changes. It is certain that foil-wrapped chocolate is less affected by damp atmosphere; indeed, well-wrapped

chocolate is entirely resistant to damp, though the adverse conditions of heat, often accompanying dampness, seem to cause some deterioration, especially if applied continuously, or intermittently, over long periods of time. It may, therefore, be asserted that wrapping chocolate in "silver paper" or foil is beneficial to the producer and to the consumer; to the latter, also, because the chocolate is maintained in more hygienic condition.

During the war, many substitutes for "silver paper" were tried, waxed paper and other moisture-resisting papers, but, from examination of the chocolate wrapped in these materials, it is certain that they were less effective in keeping the chocolate in good condition than tin foil. Before the war, aluminium foil was very popular, not only because it was slightly cheaper than "silver paper," so far as its covering capacity was concerned, but owing to the ease with which it could be embossed or printed with fancy designs and colours. In appearance, it made a very attractive wrapping, but, owing to the fact that it was full of minute holes, aluminium foil was far less effective than "silver paper" in keeping the chocolate in good condition. As both "silver paper" and aluminium foil are sold by the pound weight, the latter being very much lighter than the former though higher in price, a pound of aluminium foil contains very many more sheets than does a pound of "silver paper" which, though cheaper by weight, has less covering capacity. It becomes, then, a matter for the manufacturer to decide which foil to adopt from the economic point of view, though, as has been indicated, there is no doubt which is the more efficient.

From the point of view of the consumer, who often imagines that wrapping is a means of enabling the manufacturer to sell paper at the price of chocolate, it should be emphasised that the matter has been discussed on many occasions. Whilst, weight for weight, tinsel or foil is more expensive than chocolate, some retailers have expressed the

opinion that their customers would prefer to receive the extra chocolate for their money. Others rightly hold that the wrapping preserves the chocolate from contamination in handling and creates a special demand for chocolate attractively wrapped and protected. Again, some tablets are so thin that they break very easily, with the result that, if they were not wrapped, loose, broken chocolate would have to be sold.

The selection of the wrapping material is important, not only for those reasons already mentioned, but also because chocolate readily takes up odours, pleasant and otherwise, from outside sources or materials with which it comes in contact. Air-tight wrapping thus prevents contamination by outside odours and, also, preserves the naturally delicate aroma of the chocolate. On the other hand, carelessly selected wrappers, smelling of substances used in their preparation, are worse than no wrapping at all, since the chocolate, so wrapped, would taste of the paper. The same remarks apply to cardboard and wood containers, which readily impart to the chocolate their own characteristic taste or smell. There is so much chocolate, spoiled in this way, on the market, at the present time, that the emphasis which has been laid here upon the nature of the wrapping must not be considered to be misplaced.

In America, in 1910, the Board of Food and Drugs Inspection collected information on the subject of protecting chocolates and other confections with a coating of shellac or other gums. This coating was applied for the same purposes as wrapping in foil—to protect the chocolate against damp and the subsequent unsightliness. The Board came to the decision that it was an undesirable practice, on the ground that it was detrimental to the consumer, but it is a significant fact that, in recommending the discontinuance of the practice of dipping the chocolates in gum or weak alcoholic solutions, the Board advised confectioners against the exposure of

chocolates and other sweets in their shop windows for any lengthy period.

It is not intended to give an account of wrapping machines for covering finished chocolates with paper and foil. Such machines can now be obtained in any country, though America has specialised particularly in this branch of engineering.

#### "BLOOM."

The "bloom" that appears on chocolates, usually after they have been kept for some time, is similar in appearance to that of the "bloom" on grapes—a soft bluish-white efflorescence showing on the surface of the chocolate. The occurrence of this "bloom" has been attributed to various causes—to the fat, sugar, nature of the covering-chocolate used, and to the conditions under which the chocolates have been kept. We have considered this matter very closely, and we have not been able to find any make of chocolate which is not subject to this defect under conditions of fluctuating temperatures. Firstly, it occurs on bitter chocolate or cacao mass, hence the sugar theory may be dismissed, and it will occur on most chocolates allowed to cool very slowly at normal room temperatures. It occurs more often on very fatty chocolates, and, hence, is liable to be found most commonly on chocolate-covered goods, such as chocolate biscuits, fancy chocolates, etc. Secondly, stale stock of either of these kinds will often exhibit "bloom," and microscopical examination shows that it is due to minute crystals of fat. The occurrence of "bloom" is, then, more easily understood if it is appreciated that it arises on the surface of chocolates which have undergone fluctuations of temperature above and below the melting-point of the fat. It is true that dampness encourages "bloom" and that foil-wrapped goods show less "bloom" than the unwrapped article, but we have been unable to secure exactly the same appearance as "bloom" by submitting chocolates to damp air for even long periods, and there is every reason to believe that close contact of the foil with the chocolate would, to a large extent, prevent the formation of crystals of fat, at any rate, from standing out from the surface and giving the characteristic appearance which is known in the trade as "bloom."

"Bloom" can be avoided in the factory by quickly cooling the goods as soon as covered, and by not allowing the chocolate-covered articles to stand about in a warm room once they have been set. The stock-room should be kept at a uniform medium temperature, well below the melting-point of the fat, and, if shopkeepers paid the same attention to the keeping of their own stock, there would be less trouble arising between manufacturer and retailer owing to the deterioration of chocolate due to this cause.

## CHAPTER XVIII

### ANALYSES OF PLAIN EATING-CHOCOLATES AND CHOCOLATE POWDERS

PLAIN chocolates, the preparation of which has been described in the foregoing chapters, should consist only of roasted and shelled cacao beans, sugar, small quantities of flavouring matters and added cacao butter.

Booth has frequently pointed out the need for a definition of chocolate in England and has strongly advocated that the addition of starch and fats, other than those naturally present in the cacao bean, and of shell powder, should be absolutely excluded from any article which is to be sold under the name of "Chocolate."

The limit of 65 per cent. of sugar has also been suggested, but, at present, there is no legal standard for chocolate. The analyses of several well-known brands are, perhaps, the best guides as to what chocolate should be.

The quality of a chocolate does not, however, entirely depend upon its chemical composition, but largely, also, upon the nature of the bean used and the processes adopted, during manufacture. The degree of fineness, to which the chocolate has been ground, is of considerable importance at the present time, since there is, in Europe at any rate, the demand for a pleasant smoothness of consistency.

On the other hand, were it not for the careful attention given to chemical analysis, the public might still be suffering in the same way as in the days of Hassall, though it would seem that there is the greater need for definite legislation on the subject now, as the methods of adulteration are far more subtle than in Hassall's time.



In reading Hassall in "Food and its Adulteration," published in 1876, one cannot fail to be struck by the amazing frauds that existed at that time, if all he writes be correct, and of the remarkable improvements in the nature of our foodstuffs made in the last forty-five years. In quoting from the work of Normandy, he says: "Unfortunately, however, many of the preparations of the cocoanut (cacao bean), sold under the names of chocolate, of cocoa flakes and of chocolate powder, consist of a most disgusting mixture of bad or musty cocoanuts, with their shells, coarse sugar of the very lowest quality, ground with potato starch, old sea-biscuits, coarse branny flour, animal fat (generally tallow or even greaves). I have known cocoa powder made of potato starch moistened with a decoction of cocoanut shells and sweetened with treacle; chocolate made of the same materials with the addition of tallow and of ochre. I have also met with chocolate in which brickdust or red ochre had been introduced to the extent of 12 per cent.; another sample contained 22 per cent. of peroxide of iron, the rest being starch, cocoanuts with their shells and tallow."

"Messrs. Jules Garnier and Harel assert that cinnabar and red lead have been found in certain samples of chocolate, and that serious accidents have been caused by that diabolical adulteration."

Hassall, himself, from the examination of twelve samples, found added starch in eight of them, ranging from 10 per cent. of sago starch to 30 per cent. of wheat flour, besides a large proportion of sugar. He says: "Besides the above ingredients, several of the chocolates contained coloured ferruginous earths," and admits that, as chocolate is a compound article, there can be no valid objection to the inclusion of sugar and starch, provided that the composition is acknowledged, the main points to be considered being the price of the article and the proportions and quality of the ingredients of which it is composed.

It is safe to say that, in these days, with the exception of chocolate powders and the very cheapest eating-chocolates, and, occasionally, covering-chocolates, even the admixture of starch is unknown, whilst animal fats and added mineral matters are never met with as adulterants.

Shell powder or husk is a fairly common adulterant still, and cacao butter substitutes may be frequently found in chocolates, though it is seldom that the latter are employed unless it is required to harden, as well as to cheapen, the material.

The majority of books, quoting the adulterants of cocoa and chocolate, still include, besides sugar and starch, Venetian red, cinnabar, brickdust and peroxide of iron, oil of almonds, coconut fat, beef and mutton fat. With the severe competition among chocolate manufacturers of the present day, it would be madness to make use of the mineral matters, enumerated, for colouring purposes or loading, as their presence, if detected, would condemn for ever the products of the factory from which such chocolate emanated. The author knows, however, of cases in which lamp black has been used in order to obtain dark chocolate, in demand in certain markets.

Oil of almonds would not be added as an adulterant, firstly, because of its high price, secondly, because of its low solidifying-point, and, thirdly, because, in the best French and some English chocolates, a small quantity of powdered almonds or hazel nuts is included to produce characteristic flavour, and their presence cannot, in any way, be looked upon as adulteration, if small quantities of harmless flavouring matter are permissible in chocolate.

Coconut fat may possibly be found in some chocolates, and it is probable that its inclusion will become more general, if the increasing market value of cacao butter continues. In itself, coconut fat is harmless and pleasant, but, in chocolate, it is undoubtedly an adulterant. The melting-

point of coconut fat is somewhat lower than cacao butter, and it does not possess the same body. It would, therefore, be included for no other purpose than cheapening the chocolate, as it would not impart hardening qualities.

Beef and mutton fat are not likely to be found in chocolate, as the combination of animal fats and cacao butter do not keep well, and, after the chocolate containing either of these animal fats has remained in stock for a short time, it will acquire a tallowy taste which will predominate over the aromatic flavour of the cacao.

It is, however, desirable to be prepared to meet with any possible adulterant, and the methods of detection of any added matter found in chocolate, to date, will be discussed in the third part of the book.

In the quarter ending December, 1908, the Borough Analyst to the Southampton Town Council examined seven samples of chocolate and, in his remarks on same, said that this article was a mixture of cocoa with starch, sugar, and other materials, varying in price from at least 2s. to 8d. *per* pound retail, the difference in price depending upon the quality and relative proportions of the materials. The samples received were all of the cheapest quality, retailed to the poorer classes at two ounces a penny. Their constituents were cocoa, sugar, wheat-starch, coconut in two cases, and, in four cases, condensed milk had been used. They were all free from oxide of iron which was sometimes used to deepen the colour in the cheaper kinds. One of the samples of milk chocolate was distinctly rancid, owing to slight decomposition of the constituents, and would be liable to produce disturbance of the digestive organs, if partaken of freely. With that exception, they were all good value for money and healthy heat-giving foods.

Booth \* has made a careful study of the analysis of

\* N. P. Booth, *The Analyst*, 1909, xxxiv., 145.

chocolates, and, as the results, obtained, are interesting and valuable, a few of them are here included :—

TABLE XL.—*Analyses of some Plain Chocolates.*

	I.	II.	III.	IV.	V.	VI.	Average of twenty-four samples.
Fat . . . . .	34.60	33.60	25.60	22.10	28.10	23.70	27.40
Sucrose . . . . .	46.00	50.50	53.00	54.00	52.20	54.00	53.40
Nitrogen . . . . .	0.86	0.81	0.86	0.92	1.14	1.05	0.93
Mineral matter . . . . .	1.45	1.05	1.41	1.22	1.50	1.48	1.32
Cold-water extract . . . . .	50.00	54.80	56.90	58.10	58.20	58.90	56.80

These figures show, merely, the fluctuation in the composition of chocolate, due to the presence of different quantities of added sugar and cacao butter, and are typical of the majority of better-class chocolates on the market.

The same author has, however, analysed several adulterated chocolates, with the following results :—

TABLE XLI.—*Analyses of some Adulterated Plain Chocolates.*

	I.	II.	III.	IV.	V.	VI.	VII.	VIII.
Moisture . . . . .	1.80	0.80	1.10	1.20	3.03	1.20	3.80	3.60
Mineral matter . . . . .	1.04	1.01	1.03	1.96	2.13	0.85	0.88	1.05
Fat . . . . .	25.90	23.00	23.30	30.70	32.93	29.80	27.20	24.35
Sucrose . . . . .	48.00	56.00	56.00	44.00	46.00	47.00	40.00	—
Foreign starch . . . . .	15.00	8.00	7.50	20.00	Nil.	10.00	20.00	—
Character of starch . . . . .	Arrow-root	Wheat	Wheat	Maize	—	Arrow-root	Arrow-root	Sago
Fat-free cocoa matter . . . . .	10.00	12.00	12.20	8.30	20.60	12.00	8.40	—
Fibre (calculated on fat-free cocoa matter) . . . . .	8.70	10.00	9.80	16.60	4.50	8.50	10.00	8.80

The high percentage of mineral matter in sample IV. is attributed to the addition of red ochre, and the 32.93 per cent. fat, found in V., consisted mainly of foreign fat.

It is impossible to fly in the face of these recorded figures and to say that all chocolates are free from adulteration of added mineral matter and foreign fats, but it is our experience that the majority of chocolates on the market are remarkably pure, with the exception of occasional addition of starch and cacao butter substitutes, which are usually of vegetable origin, and of the careless or intentional inclusion of cacao husk or shell powder.

Those chocolates with under 25 per cent. of fat were probably of the dry variety, as the large proportion of sugar present, amounting to over 55 per cent. of the chocolate, would have the tendency to dry up the existing fat and render them more suitable for the preparation of chocolate powders or for use as a beverage.

Chocolates containing 60 per cent. of sugar would be incapable of being moulded without further addition of cacao butter.

Génin \* has divided his analyses under the headings of German, French and Spanish chocolates, and his results are collected in Table XLII.

TABLE XLII.—*Analyses of some Plain Chocolates.*

	German.	French.	Spanish.
Moisture . . . .	1.88—2.50	0.98—1.28	1.20—1.50
Fat . . . . .	22.50—28.55	21.40—23.80	20.50—26.60
Nitrogenous matter . .	5.81—8.18	4.57—4.99	6.45—8.67
Theobromine . . . .	0.56—0.80	1.26—1.43	1.82—2.64
Sucrose . . . . .	37.86—55.31	56.34—59.07	41.40—54.0
Starch . . . . .	4.44—6.49	0.97—1.83	1.33—1.74
Cellulose . . . . .	0.70—2.10	—	—
Ash . . . . .	1.44—2.01	1.75—1.87	2.43—3.23

From these figures, it would appear that the French favour the driest and sweetest chocolate, the Germans preferring those more fatty. It is a pity that no figures are given for cellulose in the Spanish chocolates, as the high

\* M. V. Génin, *Encyc. Chimique*, x., 4504.

percentage of ash, therein, would indicate that there is a larger percentage of husk, included in them, than in the others. As the figures stand, and without the cellulose values, the high ash-content may be due to some treatment of the beans with alkali, or some other mineral or mineral-containing matter, for purposes of improving the flavour of the chocolate.

Smooth fatty chocolates, called in France "*chocolats fondants*," have taken the popular fancy very considerably in the last few years. This may be due to the popularity enjoyed by milk chocolates, which are smooth to the palate.

There is no secret in the composition of these chocolates, which owe their character entirely to the method of manufacture and to the extra quantity of cacao butter that they contain. The method for their manufacture is described in the next chapter.

Another important feature of the chocolates of the smooth, fatty variety is the extreme fineness to which they are ground, a condition which is only reached by continual working in a "*melangeur*" or "*conche*" machine and by frequent refining.

An analysis of "*Meltis*," manufactured by Messrs. Peek, Frean & Co., Ltd., which has been taken as typical of a high-class chocolate of this variety, is given :—

*Analysis of "Meltis"—a "Chocolat Fondant."*

Moisture	.	.	.	.	1.04 per cent.
Sugar	.	.	.	.	52.50 „
Cacao butter	.	.	.	.	28.50 „
Nitrogen	.	.	.	.	0.80 „
Ash	.	.	.	.	1.60 „
Added starch	.	.	.	.	<i>Nil.</i>
Cacao husk	.	.	.	.	<i>Nil.</i>

The cacao butter was found to be the natural fat of

cacao, free from all substitutes. Under the microscope, the sugar was seen to be in an extremely fine state of division, and the mass was entirely free from husk or added starch.

All good "fondant" chocolates show about the same analysis, the difference in the various flavours, consistencies, etc., being due to the blends of beans, used, and to the methods of preparation.

Dubois, in a private communication to the author, has submitted some analyses of American chocolates, a few of which are shown in Table XLIII.

TABLE XLIII.—*Analyses of some American Plain Chocolates.*

	I.	II.	III.	IV.	V.	VI.	VII.
Sucrose . . . . .	57.60	64.05	55.10	55.95	57.21	41.30	50.70
Fat . . . . .	25.16	21.28	28.58	23.40	23.67	35.75	34.76
Crude fibre . . . . .	1.28	0.88	0.89	2.25	1.17	1.49	—
Ash . . . . .	1.35	1.17	1.27	1.78	1.75	2.15	—
Water-insoluble ash . . . . .	—	—	0.70	0.58	1.03	—	—
Water-soluble ash . . . . .	—	—	0.57	1.20	0.72	—	—
Acid-insoluble ash . . . . .	—	—	0.08	0.23	0.18	—	—
Alkali-soluble ash . . . . .	—	—	0.50	0.95	0.55	—	—
Alkali-insoluble ash . . . . .	—	—	1.00	1.00	1.18	—	—
Moisture . . . . .	—	—	0.15	0.88	0.88	—	—
Reichert-Meißl value of fat . . . . .	—	—	0.42	1.40	1.99	—	—
Refractive index at 40° C. of fat . . . . .	—	—	—	1.4562	1.4552	—	—
Saponification value of fat . . . . .	—	—	195.60	206.30	213.60	—	—
Iodine value of fat . . . . .	—	—	—	30.80	26.10	—	—
Crude fibre on s.f.-w.-free basis . . . . .	—	—	5.50	11.38	6.41	—	—
Ash on s.f.-w.-free basis . . . . .	—	—	7.85	9.00	9.59	—	—
Water-insoluble ash on s.f.-w.-free basis . . . . .	—	—	4.33	2.93	5.65	—	—
Water-soluble ash on s.f.-w.-free basis . . . . .	—	—	3.52	6.07	3.94	—	—
Acid-insoluble ash on s.f.-w.-free basis . . . . .	—	—	0.49	1.16	0.98	—	—
Alkali-soluble ash on s.f.-w.-free basis . . . . .	—	—	3.10	4.78	3.01	—	—
Alkali-insoluble ash on s.f.-w.-free basis . . . . .	—	—	6.20	5.06	6.47	—	—

Only VI. and VII of these analyses are those of "fon-

dant" chocolates which, even at the time of the author's visit to America, late in 1919, had not found great popularity on that continent. Further, it will be observed, from the chemical and physical constants of the fat, that other fats, besides cacao butter, had been used occasionally.

In a searching investigation of Wiley, published in his "Tests of Foods and Beverages," 1914, strong criticisms were passed on many American products. Without expressing any opinion on the actual report, which is clearly that of a fanatic, the author is of the opinion that much good would result to the public if similar tactics were adopted in this country.

In the report below, the starred products were those marked and rated at 85 to 100, as representing high quality and full weight and measure, with accurate labelling and reasonably conservative claims.

"N" indicated non-committal, rating between 76 and 84, given to products subject to such criticism, "in my judgment," as incorrect labelling, slightly short-weight, relatively inferior quality, etc.

"D" indicated disapproved products, rated at 75 and less, which were, in a few instances only, actually harmful, but which were so classed because they were, "in my opinion," of low quality, were misbranded, or carried grossly exaggerated claims as to efficiency or nutritive value. Of this class, there are no examples of the specimens of cocoa and chocolate that Wiley examined.

"Baker, Walter, & Co., Dorchester, Mass.

\* Breakfast Cocoa. (Claim 'absolutely pure' untenable, as always.)

Bensdorp, Amsterdam, Holland. (Bartlett, Stephen L., Co.).

N Royal Dutch Cocoa. (Good Dutch process cocoa, but presence of added mineral ingredients makes statement 'Is acknowledged by the leading authorities



to be absolutely pure cocoa' especially objectionable.)

*Blookers, Amsterdam and New York.*

Blookers' Daaldera Cocoa. (Dutch process,  $3\frac{1}{2}$  per cent. alkali added, properly labelled, good quality.)

*Huyler's, New York.*

Cocoa, Huyler's. (Mineral ingredients slightly high; not added alkali, however.)

*Lowney, Walter M. Company, Boston, Mass.*

Breakfast Cocoa. (Good quality; extravagant claims 'Unlike any other'; 'Of special benefit to those of delicate digestion'.)

*Maillard, Henry, 35th and 5th Avenue, New York.*

Maillard's Breakfast Cocoa. (Extravagant claims as to solubility, 'absolute purity'; no cocoa is 'thoroughly soluble'.)

Maillard's Premium Chocolate.

*Peter Cailler Kohler Swiss Chocolates Co., Fulton, N.Y.*

\* Breakfast Cocoa. ('Absolutely pure' claim extravagant.)

*Phillips, Charles H., Chemical Co., New York.*

N Phillips Digestible Cocoa. (No right to the claim of special digestibility; phosphates added, about 20 per cent. of sugar and vanilla—declared on label.)

*Stollwerck Brothers, Inc., New York.*

Gold Premium Brand Chocolate.

*Van Houten and Sons, Holland.*

N Van Houten's Famous Cocoa, unexcelled. (Greatly exaggerated claims as to improvement of the product by the Dutch treatment with alkali.)

*Whitman, Stephen F., and Son, Philadelphia, Pa.*

Instantaneous Chocolate. (A sweet chocolate about half sugar, 24 per cent. fat; should be so labelled.)

*Yours Truly Company, Chicago, Illinois.*

\* Yours Truly Breakfast Cocoa. (Not 'absolutely pure,' nothing is.)

N Yours Truly Premium Chocolate. (Samples examined were coated with shellac ; no arsenic, otherwise good quality.)"

Table XLIV. shows figures, obtained in the author's laboratory, for a number of well-known chocolates. It will

TABLE XLIV.—*Composition of some Well-known Brands of Hard and "Fondant" Chocolates.*

Chocolate.	Per cent.			
	Moisture.	Ash.	Cane sugar.	Fat.
Fry's "Vnello" . . . . .	1.10	1.30	53.76	32.30
Fry's plain bars . . . . .	0.82	1.60	56.33	25.00
Fry's "Caracas" . . . . .	1.10	1.20	54.52	33.50
Cadbury's "Mexican" . . . . .	1.10	1.60	54.51	31.42
Cadbury's Vanilla . . . . .	0.80	1.16	54.76	28.60
Cadbury's "Bournville" bars . . . . .	1.40	1.20	53.76	31.60
Cadbury's plain bars . . . . .	1.00	1.70	54.76	25.23
Cadbury's "Fondona" bars . . . . .	1.00	1.20	52.33	32.60
Rowntree's "Elect" . . . . .	1.20	1.52	55.80	28.40
Rowntree's "Ed." bars . . . . .	1.80	1.80	54.51	28.20
Terry's Vanilla . . . . .	1.10	1.50	52.10	33.40
Terry's "Dessert" . . . . .	1.12	1.50	51.88	33.30
Terry's "Bitter Eating" . . . . .	1.70	2.00	45.11	32.60
Terry's "Chocolat du Buffet" . . . . .	1.20	1.50	54.41	33.80
Peck Frean's "Meltis" . . . . .	1.04	1.20	52.50	28.50
Watford "Delecta" . . . . .	1.50	1.60	51.88	32.90
Watford Bousellier's plain bars . . . . .	1.50	1.40	54.51	32.50
Clarnico Vanilla . . . . .	1.60	1.52	52.33	29.30
Payne's plain bars . . . . .	1.20	1.50	54.51	29.30
Cailler's plain bars . . . . .	1.30	1.90	45.10	36.00
Kohler's plain bars . . . . .	1.20	1.40	55.54	30.00
Caley's plain bars . . . . .	1.40	1.60	50.32	33.00
Mazawattee Plain . . . . .	1.90	1.40	49.06	29.80
Barker & Dobson's Plain . . . . .	1.30	1.10	55.21	36.80
Murray's "Nuveo" . . . . .	1.46	1.72	48.65	32.20
Pascall's "Furzedown" . . . . .	1.40	1.31	56.88	33.70
Lindt's Vanilla . . . . .	0.80	1.40	49.26	29.60
Chocolate "La Palme" . . . . .	1.60	1.40	44.26	38.90
Needler's Plain Block . . . . .	0.90	1.38	52.10	35.50
Neilson's Vanilla . . . . .	1.00	1.20	51.87	32.80
Chocolat de Paris . . . . .	1.40	1.40	44.43	39.00
Rockwood's "Oriental" . . . . .	1.50	1.40	55.61	26.20
Faulder's Plain . . . . .	1.50	1.40	49.26	29.60

be noticed that there are only very small differences in the sugar- and fat-content of the different classes of chocolate. Microscopical examination, however, reveals very great differences in the fineness of the sugar, and, again, it is desired to point out that the individuality of the „special chocolates depends upon the quality of beans, used, and upon the method of manufacture.

It has already been stated that, during the war, Britain became the dumping ground for inferior chocolates. Many of the products were most objectionable to taste, to smell and to look at, yet they had a certain sale at the time. It is unfortunate that more full analyses were not conducted on the many samples that came for examination into our laboratory, but there were more important matters on hand. The few figures that follow in the next table, and the comments made, at the time, upon some of those chocolates, will show, to some extent, to what the British public submitted in the way of chocolates during the chocolate-famine years of the war. The first chocolate is of American manufacture. The second Dutch, and the third and worst British, and these are only three selected from a very large number of similar concoctions.

	I.	II.	III.
Moisture per cent. . . . .	1.22	0.86	9.70
Ash per cent. . . . .	0.76	0.50	2.06
Cane sugar per cent. . . . .	57.06	60.00	20.14
Glucose per cent. . . . .	—	—	12.86
Fat per cent. . . . .	33.13	32.18	37.74
Crude fibre per cent. . . . .	—	—	5.54
Butyro-refractometer value of fat at 40° C. . . . .	44.70	45.00	44.50
Melting-point of fat in degrees C. . . . .	26.00	28.30	29.00

I. The flavour was most objectionable and could have been obtained only from the most inferior beans. The sugar particles, extracted with dry ether, showed large pieces  $10-15$  mm. in size and appeared to be only slightly, if at

all, refined. The cacao mass was not fine and contained large quantities of husk. The fat was largely cacao butter substitute of low melting point.

II. Most unpleasant to the palate, suggesting cacao husk and flavouring matter. The sample melted easily, and, when the fat was extracted and the ether driven off, two portions, one liquid and one solid, separated out in the proportion of 1 to 2, respectively. The chemical and physical constants of the extracted fat suggested a mixture of one part palm kernel oil to two parts of cacao butter.

III. Extremely unpleasant to taste, very gritty under the teeth and much inferior to any of the common brands of genuine chocolates, examined previously. Largely composed of cacao husk, sugar, syrup, cacao butter substitutes and starch.

#### Chocolate Powders.

Chocolate powders, such as many makers now produce, usually contain from 20—25 per cent. of fat and 50—60 per cent. of sugar, with the addition of foreign starch, in the majority of cases, to add to the thickening property of the beverage and to the maintenance of the chocolate in a fine, powdered form.

Ewell \* gives the following analyses of some commercial chocolate powders :—

TABLE XLV.—*Analyses of some Commercial Chocolate Powders.*

	Rowntree's powdered chocolate.	Epps's prepared cocoa.	Fry's Diamond sweet chocolate.	Chocolate Menier.
Fat . . . . .	25.84	25.94	18.60	21.31
Fibre . . . . .	1.30	1.51	0.81	1.10
Sucrose . . . . .	51.00	26.00	55.00	58.00
Ash . . . . .	1.66	3.15	1.16	1.40
Added starch . . . . .	Very small amount of arrowroot.	Much arrowroot.	Much wheat starch, some arrowroot.	Nil.

\* E. E. Ewell, *U.S. Dept. of Agriculture, Bull. 13.*

Bell \* has given analyses of several preparations of cocoa, such as "Iceland Moss Cocoa," "Rock Cocoa,"

TABLE XLVI.—*Analyses of some American Chocolate Powders.*

	Swiss milk cocoa.	Ground chocolate.	Ground chocolate.	Powdered sweet chocolate.	Cup chocolate.
Moisture . . . .	—	1.15	0.65	—	1.43
Sucrose . . . .	11.80	52.36	57.77	41.08—54.46	44.14
Fat . . . .	24.68	15.28	18.90	19.95—25.04	14.05
Crude fibre . . . .	—	2.02	1.40	—	0.87
Ash . . . .	5.40	2.80	1.93	—	3.16
Water-insoluble ash . . . .	1.80	0.75	1.30	—	2.24
Water-soluble ash . . . .	3.58	2.05	0.63	—	0.92
Acid-insoluble ash . . . .	—	0.09	0.11	—	0.00
Alkali-soluble ash . . . .	—	1.80	0.50	—	0.35
Alkali-insoluble ash . . . .	—	1.65	1.70	—	3.05
Reichert-Meissl value of of fat . . . .	2.35	0.29	0.32	—	0.45
Saponification value of fat . . . .	—	194.90	195.4	—	196.7
Iodine value of fat . . . .	—	—	—	—	—
Crude fibre on f.-s.-w.- free basis . . . .	—	6.47	6.17	—	—
Ash on f.-s.-w.-free basis . . . .	—	8.98	8.51	—	—
Water-insoluble ash on f.-s.-w.-free basis . . . .	—	2.41	5.73	—	—
Water-soluble ash on f.-s.-w.-free basis . . . .	—	6.57	2.78	—	—
Alkali-soluble ash on f.-s.-w.-free basis . . . .	—	5.80	2.20	—	—
Alkali-insoluble ash on f.-s.-w.-free basis . . . .	—	5.30	7.50	—	—
Acid-insoluble ash on f.-s.-w.-free basis . . . .	—	0.29	0.48	—	—
Lactose . . . .	1.50	—	—	—	13.20
Butter fat . . . .	1.95	—	—	—	—
Microscopical examina- tion. . . .	Trace of wheat starch.	—	—	—	—
Casein . . . .	Trace	—	—	—	9.99

"Flake Cocoa," and "Chocolat de Santé," which consisted of 2.5–5.5 per cent. of moisture, 17–25 per cent. of fat, *nil* to 25 per cent. of added starch, and

\* J. Bell, "The Chemistry of Foods," 1887, 89.

*nil* to 61 per cent. of added sugar. These preparations are seldom met with now and are of little or no commercial importance.

More recently, Dubois has sent the author the above analyses, shown in Table XLVI., of some American chocolate powders which include some containing milk solids.

## CHAPTER XIX

### RECIPES FOR MAKING CHOCOLATE.—PLAIN AND “FONDANT” CHOCOLATES, AND CHOCOLATE POWDERS

FROM the analysis of a chocolate, it is very easy to reconstruct one possessing the same chemical composition, but it is exceedingly difficult, without much experience, to determine how a special flavour or aroma is obtained.

This is obvious, seeing that different methods in manufacture will give different aromatic qualities to the cacao and that small quantities of an essential oil, blending with the natural aroma of the cacao, will completely alter the flavour of a chocolate.\*

Treatment with alkali will give a certain result, and chocolate, so prepared, can be detected by analysis, but the blending of different varieties of beans, which is of high importance in chocolate-making, and the addition of a drop or two of an essential oil to a hundredweight of chocolate, cannot be so determined.

Various flavours, such as vanilla, cinnamon, and other spices, coffee, oil of coriander and other aromatic bodies, combine so well with cacao that nearly every manufacturer of chocolate has some secret condiment, or blend of flavouring matters, with which to give his products a character, and which, he claims, secures a unique result.

Each country may be said to have its fancy in flavour : France prefers its chocolate mixed with almonds, hazel-nuts or chestnuts in small quantities, to give a nutty flavour, Spain likes spiced chocolate, America consumes mostly milk

\* *Vide* Chapter XXIII.

chocolate, often with whole nuts or almonds embedded in the cakes, while England favours vanilla flavour. Every country, moreover, has a slightly different variety of beans from which to select, and the characteristic flavours of the varieties of cacao beans, a subject already dealt with, play no small part in determining the final flavour of the resulting chocolate. Added to this, special methods of preparation, particular to individual factories, enable an almost endless number of combinations to be made.

It is not intended to give a very long list of possible blends of cacao, for it would be useless, but a few selected recipes of known worth have been chosen.

### Dry Chocolates.

#### FRENCH RECIPES.

Recipe 1. (*Emile Duval in "Confiserie Moderne."*)

	lbs.	ozs.
Arriba . . . . .	2	3
Para . . . . .	3	4
Machala . . . . .	2	3
Large nibs . . . . .	20	12
Almonds . . . . .	1	2
Hazel-nuts . . . . .	4	6
Cacao butter . . . . .	1	2
Chestnut sugar . . . . .	38	4
Ordinary sugar . . . . .	16	0
Vanillin sugar . . . . .	0	2
Ordinary sugar (second addition)	10	10
	<hr/>	<hr/>
	100	0

The figures are somewhat awkward, but they have been obtained from an original recipe and increased to total 100, so that estimation of the percentages of cocoa, fat and sugar can be readily made.

The chestnut sugar in the above recipe is prepared by



boiling the waste syrup, after the chestnuts for "Marrons Glacés" have been crystallised in it, to  $145^{\circ}\text{C}$ .; the sugar is then briskly stirred, so as to cause granulation and a sandy consistency ("sablé"), and dried in a drying room prior to its addition to the chocolate. This sugar, which, owing to its dark colour, has no other application in confectionery, can, with advantage, be used in chocolate for its nutty flavour.

The chocolate from this recipe is one of the dry variety, and for this reason, the whole of the sugar should not be added at once, but, after the first portion has been worked into the cacao paste in the "mélangeur," the mixture is placed in a pan in the hot cupboard, to recover its softness. The rest of the sugar is then added, and the whole well worked again in the "mélangeur," refined to the required smoothness and moulded. This is not a chocolate of finest quality.

#### Water Chocolates.

Whilst speaking of chocolates of the dry variety, it is impossible to pass over the mention of so-called "Water Chocolates." Manipulation with water, to which manufacturers occasionally resort, is for the purpose of facilitating the working of the chocolate paste in the process of manufacture and moulding. Chocolates that contain only small quantities of fat, say below 20 per cent., are, clearly, more of a doughy than of a liquid consistency and, consequently, will not stand close refining and are moulded only with difficulty. It is not correct to say that such chocolates are fraudulent, yet, again, the public does not get what it expects when "water chocolates" are palmed off on it. The "Verband deutscher Schokoladen Fabrikanten," at one time, took up a severe attitude towards the use of fat-saving materials, especially gum tragacanth and glue, for the detection of which Welmans devised various methods of analysis. As one journal has tritely put it, "Since glue has been abandoned, and the same results have been got with unadul-

terated pump water, no one concerns themselves about the matter." Yet, again, this criticism is hardly fair, since no appreciable quantity of water can be left in the chocolate without rapid deterioration of the product.

An inquiry into "Water chocolates," put on foot by the *Gordian*, resulted in a correspondence to which this journal contributed some amusing criticisms. The letter from Herr Vogel of Dresden is quoted as follows :—

"In reply to your favour of the 2nd inst., I beg to say that so far as I know the addition of water in chocolate mixing is only very small, and is only made when the sugar is unusually dry. We have here in Germany sugar makers who make a very inferior sugar which is much used for cheap chocolates. This brand of sugar has a certain dampness of its own, which makes it suitable for chocolate manufacture. There are, however, some sugars which, especially when finely ground, are so dry that they must be watered for amalgamating purposes. Only a very little water is necessary to supply the moisture which most sugars have already. I cannot see that such an addition is unpermissible, let alone punishable. If too much water is used it would be adulterating. At the same time I cannot think that more water would be added than could be helped, because it would have to be evaporated off again before the chocolate was fit for moulding, and at the usual temperature of mixing this would mean much waste of time."

The letter closes with a promise to have some fat determinations made.

After saying that the letter had been put among the archives, *Gordian* firstly states that it has never heard that moist sugar, sugar with a "certain dampness," was better than dry sugar for chocolate-making, and that it never knew that dry sugar had to be damped before a chocolate mass could be made with it. *Gordian* then remarks, with philosophic calm, that every trade has its own methods and its

own experiences, and adds that it once had a block chocolate under notice that was as hard as stone, which, as it contained only 18 per cent. of fat, must have consisted of 70 per cent. sugar, 2 per cent. butter, and 28 per cent. cocoa. A mass with such a composition could not be made in the usual way so as to be suitable for moulding.

There is no doubt, however, that sugar containing a certain very low percentage of moisture is better for making chocolate than the bone-dry, finely pulverised crystal, if a simplification of the refining process and a saving of cacao butter are to be considered. The recent work of Bradley and others has put the point beyond a shadow of doubt, and the use of water in the manufacture of chocolate, for the purpose of conserving cacao butter, is common knowledge. The patents covering the manufacture of "amorphous sugar," to which reference has already been made, also emphasise this point, but, as in all processes, there are limits beyond which it is undesirable to go. The presence of 1—2 per cent. of moisture in the sugar, which subsequently dries out till only some 0.5—1 per cent. is left, in order to simplify refining processes and to make a small conservation of cacao butter, is a very different matter to adding large quantities of free water to a dry chocolate mass in a "mélangeur," in order to bring the total percentage of cacao butter below twenty. The fact still remains, also, that "Water chocolates," as ordinarily prepared, are inferior chocolates for eating purposes, abnormally low in fat and of inferior flavour.

"Water chocolates" may, however, be used for making the beverage "Chocolate" which requires only a small amount of fat to be present, and many makers manufacture this quality of chocolate for this purpose, stating clearly on the labels of the packets the uses to which the contents should be put.

In order to conform with the suggested standard for

sweetened chocolate, (see Appendix) a minimum of 19 per cent. of cacao butter is indicated, so that chocolates containing one more per cent. would fall within the definition. And that is all that can be said in their favour.

**Dry Chocolates** (*Continued*).

*Recipe 2.*

Caracas.	.	.	.	.	18 $\frac{3}{4}$ lbs.
Maragnan	.	.	.	.	18 $\frac{3}{4}$ "
Hazel-nuts	.	.	.	.	3 $\frac{1}{4}$ "
Sugar	.	.	.	.	59 "
Vanilla	.	.	.	.	$\frac{1}{4}$ "
					<hr/>
					100 lbs.

*Recipe 3.*

Caracas.	.	.	.	.	16 $\frac{1}{4}$ lbs.
Para	.	.	.	.	9 $\frac{1}{4}$ "
Trinidad	.	.	.	.	18 $\frac{1}{2}$ "
Sugar	.	.	.	.	55 $\frac{3}{4}$ "
Vanilla	.	.	.	.	$\frac{1}{4}$ "
					<hr/>
					100 lbs.

It is needless to enumerate more of such recipes, the last mentioned being typical of the best chocolates of the dry variety. If such a chocolate were well milled, it could not be surpassed, the blend of Caracas, Para and Trinidad beans being ideal for flavour, whilst the percentage of sugar is not too high.

GERMAN RECIPES.

*Recipe 1.*

Cacao mass	.	.	.	.	49 lbs.
Sugar	.	.	.	.	49 "
Cinnamon	.	.	.	.	1 $\frac{1}{2}$ "
Vanilla	.	.	.	.	$\frac{1}{2}$ "
					<hr/>
					100 lbs.

*Recipe 2.*

Cacao Mass . . . .	39 lbs.
Sugar . . . . .	59 "
Cinnamon . . . . .	1½ "
Cloves . . . . .	½ "
<hr/>	
	100 lbs.

The cacao mass should consist of the better quality beans, such as Caracas, Arriba, or Puerto Cabello, for the finest chocolate, though each maker has his own secret blend.

Zipperer gives a list of several working formulæ, such as one part each of Caracas and Guayaquil, one part each of Maracaibo and Maragnan, one part of Caracas to five parts Maragnan, and so on.

In the second recipe shown above, it is certain that extra cacao butter would have to be added to facilitate working, as the large proportion of sugar would tend to dry the paste.

## SPANISH RECIPES.

*Recipe 1.*

Puerto Cabello . . . .	26 lbs.
Caracas . . . . .	18 "
Cuba . . . . .	12 "
Sugar . . . . .	44 "
Cinnamon . . . . .	to flavour
<hr/>	
	100 lbs.

Jacoutot, speaking of this recipe, says : " Spanish chocolate is roasted in a special manner. It is its imperfect roasting which gives it the peculiar strong flavour which distinguishes it." The same author recommends that the cacao be first washed in warm water and then boiled with the sugar and sufficient water, till the sugar rises in the pan. The pan is then removed, and the whole grained in the usual manner, as for " Praliné." The graining mass is loosely

powdered, and dried or worked in the "mélangeur" and refiners, till free from moisture.

The author has seen chocolate made in Spain with raw sugars, and, undoubtedly, they often owe their fine flavour to this ingredient.

Such a process as that given by Jacoutot is, however, laborious, and good results can be obtained by using half-roasted cacao and treating in the same way as for ordinary chocolate, with a subsequent heating in a "conche" machine.

*Recipe 2.*

Cacao mass	.	.	.	.	48 lbs.
Sugar	.	.	.	.	51 $\frac{3}{4}$ "
Spices	.	.	.	.	$\frac{1}{4}$ "
					<hr/>
					100 lbs.

The spices are composed of three parts of cinnamon, one part cloves, two parts cardamoms, one part mace, and one part vanilla.

For other recipes of plain eating-chocolates, it is suggested that works on confectionery be consulted, the only purpose served by including here those recipes, already given, being to show the possibility of obtaining good-flavoured chocolate by special blends of beans and aromatic spices and to give the proportions of sugar to cacao that are sometimes employed.

**Alkalised Chocolate.**

Chocolates which have been treated with alkali, during the roasting of the beans or in the process of manufacture, should, perhaps, be included under the heading of "plain." In the former case, the method of procedure is the same as for rendering cocoa powder "soluble," whilst, in the latter case, very small quantities of a strong solution of caustic soda or potash are added to the chocolate paste while running

in the "melangeur" and are, with advantage, added prior to the first refining. If the chocolate is so treated, the greatest care should be taken to free it from moisture before moulding, and, consequently, the smallest possible quantity of water should be employed for making the alkaline solution.

In some factories in Switzerland and Germany, the chocolate is treated with alkali, as it is believed that the flavour is improved thereby. This, however, is a matter of some difference of opinion, and it is our experience that the chocolate of finest flavour is prepared by using the best quality beans, properly roasted, without any further treatment.

In those factories where alkali is employed, the caustic soda or potash is dissolved in the minimum quantity of water and added to the chocolate paste in the proportion of  $\frac{1}{2}$  lb. to  $\frac{3}{4}$  lb. alkali to 100 lbs. of cacao paste. It is sometimes the custom to add the alkali to the cacao mass before the addition of the sugar and to dry off the moisture in a vacuum mixer, when the sugar and other matters are added at a later stage. In either case, after the paste has been dried out, the method of procedure is the same as for ordinary chocolate.

#### **"Fondant" Chocolates.**

Since the introduction of milk chocolates, the smoothness, characteristic of them, is expected by the public in plain chocolates. The dry, hard chocolates are not so popular as the smooth, fatty varieties ("chocolate fondants") with the general public at the present day.

To meet the requirements, chocolate manufacturers have been compelled to introduce a confection containing 30 per cent. and upwards of cacao butter to replace the dry chocolates of 25 per cent. fat-content. To this end, cacao butter is added to the recipes of the older-fashioned, drier choco-

lates, and the chocolate mass is refined to a much greater extent than formerly. This means, of course, that the percentage of cacao mass is reduced and that free cacao butter is added in its stead.

The "conche" machine, described later when dealing with milk chocolates, usually replaces the "mélangeur" for finishing these smooth, fatty chocolates, and the mass, after being closely refined, is worked from thirty to forty hours consecutively, sometimes for a longer time, to acquire the characteristic "fondant" nature which is so pleasant to the palate.

Swiss chocolate manufacturers, whose work is simplified by the hydraulic power at their command, run the machines day and night, carrying the manufacture of "chocolat fondant" to the end without any interruption. This continuous process gives the consistency, whilst the extreme fineness, resulting from close refining, produces the smoothness, both characteristic qualities of this class of chocolate.

"Chocolats fondants" are, of course, more costly to produce, owing to the additional amount of labour and power, expended and to the larger amount of high-priced cacao butter, used in their preparation.

The cacao butter, employed, is purchased on the market and is the outcome, or by-product, of factories where cocoa powders are more largely produced than chocolate, in the preparation of which the surplus cacao butter, expressed in the manufacture of cocoa powder, could be employed. The fatty eating-chocolates more nearly approach in composition those used for covering various sweetmeats and biscuits, though, for the tablet chocolates, greater care has to be taken that the finest varieties of bean are used and that refinement is carried to the furthest degree.

To give but one example, from an infinite number possible, of methods of manipulation in order to secure a certain flavour the following description will enable the



manufacturer to obtain a chocolate with a flavour more or less closely resembling that associated with the name of Lindt, according to the care with which the process is carried out.

#### MIXING.

Arriba, or fine Venezuelan nibs	. 10 parts.
Ceylon, or Trinidad nibs . . .	. 10 "
Cuban, Bahia, or San Thomé nibs	. 10 "
Sugar . . . . .	. 70 "
Cacao butter . . . . .	. 15 "
Vanilla . . . . .	. $\frac{1}{5}$ "

The beans should be roasted only just sufficiently to enable the husks to be removed easily on the winnow. A temperature of 100° C. is sufficient for this purpose, and a temperature higher than 130° C. should not be allowed. The lightly roasted nibs are ground, preferably on granite rollers, mixed with sugar, refined on steel refiners, and then, after cold blocking, the hard blocks are levigated on granite refiners. A second refinement on steel refiners is advantageous.

Between each refining, the mass is run in a "mélangeur" for 10—15 minutes, and some cacao butter is added on these occasions. After two or three grindings on the steel refiners, followed each time by further levigation of the cold blocks on the granite rollers, the chocolate mass is placed in a "conche" machine and heated from 80° to 90° C. for seventy-two hours. By these means, the semi-roasted nibs develop their full flavour in the later stages of manufacture, and the heat, applied in the "conche," also tends to caramelise the sugar, slightly.

The "caramelising machine" of Messrs. Savy Jeanjean et Cie. aimed to produce the same effect, and, though undoubtedly the sugar is caramelised, the resulting chocolate is by no means of the same fine flavour that is produced by the slower process. The makers of this machine, however,

claim smoothness and homogeneity, rather than enhanced aroma, for chocolate treated in this way.

### Chocolate Powders. •

As distinct from cocoa, chocolate powders are, for the most part, put upon the market as a cheap cocoa preparation for drinking purposes.

In the previous chapter, some analyses of chocolate powders have been shown, and the addition of starch, besides that of sugar, common to eating-chocolate, has been noted.

The addition of starch cannot be condemned, if the public are fully aware that they are purchasing that commodity, as well as cocoa and sugar, when they ask for chocolate powder. In fact, the beverage prepared from such a mixture is most palatable, if good quality beans have been used, as it can be made to the thickness of "blanc-mange," if required, due to the gelatinisation of the starch which the powder contains, and a feeling of repletion, valuable to the poorer classes, is experienced after the consumption of a cupful prepared with boiling water, which may not be the case when ordinary cocoa powders are used.

Epps's cocoa preparation is a good example of a carefully manufactured article with high food-value, and its composition has been already given.\* A good chocolate powder may be prepared from the following recipe, in which it is assumed that the cacao mass is prepared from fine quality beans.

#### CHOCOLATE POWDER.

Cacao mass . . . . .	20 lbs.
Arrowroot (slightly baked) . . . . .	20 "
Sugar . . . . .	59½ "
Vanilla . . . . .	½ "
	<hr/>
	100 lbs.

\* *Vide* Chapter XVIII., Table XLV.

Such a recipe will show, on analysis, only 10 per cent. of fat, and, of course, if it is desired to increase the figure, the cacao mass may be increased at the expense of the sugar, or arrowroot.

Unfortunately, we more often meet with a recipe, described as "ordinary chocolate powder," composed somewhat as follows :—

"ORDINARY" CHOCOLATE POWDER.

Cacao mass . . . . .	15 lbs.
Potato starch . . . . .	15 "
Shell powder . . . . .	15 "
Sugar . . . . .	55 "
	<hr/>
	100 lbs.

We are confident, however, that, though such recipes find their way into books on cocoa and chocolate, the powders, prepared from them, do not often appear on the market. During the war, it was a different matter, and many cases of heavily adulterated cocoa and chocolate powders were encountered.

The addition of cacao husk or shell powder to cocoa and chocolate preparations is, however, by no means uncommon, as the following extracts taken from *The Times* (June 10th, 1910) will show :—

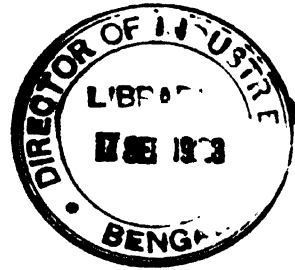
"At West London, before Mr. Fordham, W. D.— was summoned at the instance of the Fulham Borough Council for selling cocoa which contained 60 per cent. of powdered cocoa shells.

"There was also a summons against — for selling at a shop in Fulham a sample of cocoa which contained 20 per cent. of cocoa shells."

The former case was adjourned, the public analyst for Fulham being reported to have said as follows :—

"The cocoa of commerce was made of the bean deprived

of its shell. There was a certain amount of nourishment, no doubt, in the shell, but the fibrous nature of the shell rendered the article very unpleasant. He did not suggest that there was anything deleterious in the sample before the court. The shell was used for cattle food and in the cheaper form of chocolates. There was no standard in this country for cocoa."



## CHAPTER XX

### MILK CHOCOLATES AND MILK CHOCOLATE POWDERS— ANALYSES AND RECIPES—NUT CHOCOLATES, ETC.

THE popularity of milk chocolates, which probably synchronises with the introduction of Peter's milk chocolate to the public, is well deserved. Whilst it is admitted that the best milk are worthy of competing against the best plain chocolates, there are a large number of so-called milk chocolates on the market, of a glutinous and coarse nature, which cannot compare in palatability with the coarsest of the plain variety. Especially are these coarse milk chocolates to be found in America at the present time.

Booth, in a paper before the Seventh International Congress of Applied Chemistry, recommended that milk chocolate should be legally standardised as a "preparation, composed exclusively of roasted, shelled cocoa beans, sugar, and not less than 15 per cent. of the dry solids of full-cream milk, with or without a small quantity of harmless flavouring matter."

In the better brands of milk chocolate, both English and foreign, this standard is undoubtedly conformed with, but so much depends upon the fineness, to which the preparation is ground, that chemical analysis, alone, will not show the excellence of any particular brand.

Competition, however, is a wonderful leveller of quality, and, seeing that this exists in normal times to a high degree among chocolate manufacturers, competing brands of a good price show great similarity in quality.

For the cheaper brands, it is only necessary to refer to a

later page, where recipes are given for "ordinary" milk chocolates, to realise that there are greater possibilities of addition of cheap varieties of, or substitutes for, the ingredients composing them than in the case of plain chocolates. Booth,\* however, has been fortunate in his selections of samples for analysis and, in his valuable contribution to *The Analyst*, gives the following results:—

TABLE XLVII.—*Analysis of Some Milk Chocolates.*

## A.

	English.									
	I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.
Total fat . . .	30.6	32.7	36.0	23.7	27.0	35.2	29.55	35.7	34.6	33.3
Made up of—										
Milk fat . . .	7.0	2.6	6.4	2.7	4.6	4.7	8.2	8.3	3.0	7.6
Cacao butter . .	23.6	30.1	29.6	21.0	22.4	30.5	21.35	27.4	31.6	25.7
Milk sugar . . .	9.2	3.8	8.2	6.5	7.3	8.9	10.0	11.1	5.0	10.4
Cane or beet sugar	45.0	46.5	38.0	52.6	32.4	41.2	44.4	39.1	54.3	38.4
Foreign starch . .	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>
Cocoa shell . . .	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>
Nitrogen . . .	—	—	—	—	—	—	—	1.10	0.76	1.68

## B.

	Swiss, German, Austrian and Belgian.									
	XI.	XII.	XIII.	XIV.	XV.	XVI.	XVII.	XVIII.	XIX.	XX.
Total fat . . .	31.5	29.9	30.0	30.8	29.5	29.2	31.5	33.4	30.9	31.3
Made up of—										
Milk fat . . .	7.7	6.6	8.8	5.8	7.9	13.6	8.1	5.7	8.4	8.3
Cacao butter . .	23.8	23.3	21.2	25.0	21.6	15.6	23.4	27.7	22.5	23.0
Milk sugar . . .	7.5	10.0	11.0	5.2	10.8	8.4	9.0	7.5	5.5	7.7
Cane or beet sugar	36.1	45.6	42.2	35.0	52.7	36.2	42.7	42.9	50.2	42.2
Foreign starch . .	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>
Cocoa shell . . .	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>
Nitrogen . . .	—	—	—	—	—	—	1.28	1.30	1.10	—

It is not difficult to reconstruct these figures and to produce, synthetically, recipes from them, though it must always be realised that the interpretation of analyses must be accompanied by a knowledge both of the technical

\* N. P. Booth, *The Analyst*, 1909, xxxiv., 139.

methods of preparation and the qualitative, as well as quantitative, valuation of the components. Thus, taking the analysis of the milk chocolates as showing :—

*English Samples.*

	Average. Per cent.	Maximum. Per cent.	Minimum. Per cent.
Milk fat . . .	5.5	8.3	2.6
Cacao butter . . .	26.3	31.6	21.0
Cane sugar. . .	43.2	54.3	32.4

and—

*Foreign Samples.*

	Average. Per cent.	Maximum. Per cent.	Minimum. Per cent.
Milk fat . . .	8.1	13.6	5.7
Cacao butter . . .	22.7	27.7	15.6
Cane sugar. . .	42.6	50.2	35.0

a recipe, composed as follows, will comply with the above analyses in the main particulars :—

*Recipe.*

		Cacao butter.	Milk fat.	Cane sugar.
Cacao mass . . .	9.0 yielding	4.5	—	—
Cacao butter . . .	20.0 „	20.0	—	—
Cane sugar . . .	41.0 „	—	—	41.0
Vanillin sugar . . .	2.75 „	—	—	2.0
Milk powder (full- cream, 25 per cent. fat) . . .	27.25 „	—	6.81	—
		100.00	24.5	6.81
				43.0

According to the percentage of cacao mass and butter, respectively, that is to be employed, so can the one be increased at the expense of the other, always remembering that cacao mass contains some 50 per cent. of cacao butter. Further, according to whether a dark or a light chocolate is required, so can these proportions be again varied, the higher the percentage of cacao mass used the darker the chocolate.

So far as the milk flavour is concerned, 20 per cent. of milk solids of full-cream milk will usually be found to be sufficient, though, here again, if the cacao mass is also of high percentage, the flavour of the latter may be inclined to mask that of the former.

The strength of flavour of the different beans, employed, must also be considered in making a recipe, as it is quite possible to use a large quantity of mild-flavoured cacao, whereas the same quantity of a full-flavoured bean would cover the flavour of the milk. To give a list of recipes is, therefore, quite useless to the manufacturer, who must, firstly, determine which milk chocolate he fancies and then, from the complete analysis, reconstruct a recipe. The method of preparation that he adopts will, of course, play a considerable part in determining the flavour and other qualities of his chocolate.

To quote a few recipes only, in order to emphasise the difference that may be seen in the proportions of the various ingredients, the following three have been selected :—

	I.	II.	III.
	<i>Zipperer.</i>	<i>Fritsch I.</i>	<i>Fritsch II.</i>
Cacao mass . . .	28	13.25	37.00
Cacao butter . . .	12	20.50	12.25
Cane sugar . . .	36	35.00	27.00
Milk solids (full-cream) .	24	31.00	23.25
Vanilla . . .	—	0.25	—
Vanilla sugar . . .	—	—	0.50
	100.00	100.00	100.00

These, on analysis, would show approximately :—

	I.	II.	III.
Fat-free cacao mass . . .	14.00	6.68	18.50
Cacao butter . . .	26.00	27.10	30.75
Cane sugar . . .	36.00	35.00	27.00
Lactose . . .	9.25	11.90	8.95
Butter fat . . .	6.00	7.75	5.80



It will be observed that, of these recipes of Zipperer and Fritsch, only the second compares favourably with the reconstructed recipe, already given. On the other hand, it is clear that the main differences lie in the amount of cacao mass and cacao butter, present. It becomes, then, a matter entirely of taste and desire of the manufacturer who, provided he has sufficient fat, present in his chocolates, to enable him to mould it satisfactorily and to give the required consistency, is at liberty to choose whether, on the one hand, he desires a full-flavoured chocolate, in which case he selects a recipe with a large proportion of cacao mass made from mild beans or with a smaller quantity of full-flavoured cacaos, or, on the other hand, a pronounced milk-flavoured chocolate, in which case he will, probably, reduce the cacao mass and increase the proportion of cacao butter.

An analysis of a good type of British milk chocolate has been given in a number of the *International Sugar Journal*, 1902, which stated that, whilst, hitherto, the British chocolate trade had been in the hands of a few big firms, yet the manufacture of *milk* chocolate seemed to have been a monopoly of manufacturers on the Continent. Since that time, however,

*Analysis of Cadbury's Milk Chocolate.*

	Per cent
Total fat . . . . .	30.30
Cacao butter . . . . .	22.60
Milk fat . . . . .	7.70
Cane sugar . . . . .	42.00
Milk sugar . . . . .	10.90
Proteids or albuminoids . . . . .	10.30
Ash . . . . .	2.10
Moisture . . . . .	1.60
Indigestible fibre . . . . .	0.75
Undetermined bodies . . . . .	2.05
	<hr/> 100.00

the British firms have made great strides, and there is hardly one chocolate manufacturer, in this country, that does not now produce a very fair milk chocolate. The journal, to which reference has been made, gave the above analysis of Cadbury's Milk Chocolate, as it was at that time.

The alkalinity of the ash was shown as 0.16 per cent. ( $K_2O$ ).

Laxa\* has given some analyses of commercial milk chocolates, worthy of reproduction here. (Table XLVIII.)

TABLE XLVIII.—*Analyses of some Milk Chocolates.*

	Moisture.	Total solids.	Casein.	Other proteins.	Fat.	Lactose.	Sucrose.	Other non-nitrogenous bodies.	Ash.
1. Gala (Peter)	0.77	9.66	—	—	31.47	7.32	27.51	21.40	1.87
2. Delta (Peter)	0.54	9.86	5.16	4.70	30.11	8.02	48.25	20.89	2.32
3. Croquettes (Peter)	1.79	9.15	3.86	5.29	31.01	7.42	39.42	8.37	1.96
4. Milka (Suchard)	1.22	8.13	4.43	3.70	32.33	8.70	35.93	11.87	1.82
5. Croquettes (Cailler)	2.26	10.94	4.36	6.58	31.12	7.84	33.68	11.88	2.28
6. Croquettes crémant (Cailler)	0.99	6.80	0.67	6.22	31.37	2.24	33.14	23.86	1.51
7. Villards	1.00	6.83	4.46	2.37	33.12	8.00	45.22	4.28	1.55
8. Alpina (Sprungli)	0.72	9.73	3.10	6.63	33.11	8.76	37.25	8.47	1.96
9. Marnier	0.23	5.02	0.40	4.62	35.25	1.46	40.46	10.31	1.27
10. Rigi (Hartwig and Vogel)	2.03	6.98	1.43	5.55	33.67	9.14	38.87	7.82	1.49

These analyses by Laxa will not, however, stand very close investigation, if recipes are to be prepared from them.

Laxa assumes, from the estimation of lactose, that there is an undue amount of this sugar in samples 3, 4, 5, 8 and 10, a condition which he explains is due to the addition of milk sugar during condensation of the milk. This, we think, to be improbable, both owing to the high price of milk sugar as compared with sucrose or even milk powder and to the fact that we are unable to see any advantage to be derived by such addition. In the writer's opinion, the analyses of 1, 2, 6, 7 and 9 are either incorrect or incorrectly stated in Laxa's original paper.

\* O. Laxa, *Zeitsch. Nahr. Genussm.*, 1904, vii., 471.

Further, assuming that the whole of the lactose, found, be due to that naturally present in milk solids, containing 25 per cent. of fat and 38.5 per cent. of lactose, and that the remainder is pure roasted cacao mass and cacao butter (added), we obtain the following recipes, synthesised from Laxa's analyses :—

TABLE XLIX.—*Synthesis of Milk Chocolates from Table XLVIII.*

	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.
Cacao powder (fat free)	26.0	3.2	13.4	14.0	18.4	31.0	5.3	11.6	15.2	8.7
Sugar	27.5	48.3	39.4	35.9	33.4	33.1	45.2	37.3	46.5	38.9
Milk solids (or powder)	20.0	21.9	20.2	23.7	21.4	6.1	21.8	23.9	4.0	25.0
Cacao butter (total)	26.5	26.6	27.0	26.4	26.8	29.8	27.7	27.2	34.3	27.4

These results have been obtained by calculating the total amount of milk powder that would naturally contain the lactose, estimated, plus 5 per cent. for the moisture-content. If Laxa's supposition that lactose has been added during condensation of the milk be correct, the figures for milk powder in Table XLIX. for samples 3, 4, 5, 8 and 10 will be high, and, consequently, the cacao powder, estimated by difference, will be correspondingly too low.

The cacao butter is estimated by subtracting from the total fat, found, that due to the milk solids, on the reasonable assumption that the fat-content of the milk solids was 25 per cent. On the other hand, it must be realised that skimmed milk is often used, a fact which cannot be determined without the chemical and physical constants of the extracted fat.

Moreover, it must be understood that, if the amount of roasted nibs or cacao mass is to be calculated, the figures under cacao powder must be doubled at the expense of those for cacao butter, since cacao contains 50 per cent. of fat.

We have, therefore, the following percentages of cacao mass present in these chocolates : (1) 52 per cent., (2) 6.4 per

cent., (3) 26.8 per cent., (4) 28.0 per cent., (5) 36.8 per cent., (6) 62 per cent. (with a small deficiency of cacao butter), (7) 10.6 per cent. (with a large excess of cacao butter), (8) 23.2 per cent., (9) 30.4 per cent., (10) 17.4 per cent. (with a large amount of added cacao butter).

In the first edition of this work, the author criticised Laxa's figures rather severely, but, in the light of more recent work done on Milk Chocolates, it is probable that the suggestion now made is correct, namely, that only analyses 1, 2, 6, 7 and 9 are misleading.

It must be realised that the interpretation of analyses and the reconstruction into recipes, even in the hands of the expert, are full of pitfalls, as it is necessary to know so much before reconstruction can be performed with absolute certainty. Thus, during the war and the shortage of milk, other albuminoids, besides those from milk solids, were used. The necessary quantity of lactose was added to make up the deficiency of that sugar, naturally occurring, and the added fats were so adjusted as to give similar chemical and physical constants to those of a fat extracted from a normal milk chocolate. The fraud was only detected by the writer from the albuminoids and the amino acids prepared from them.

Dubois has contributed, in a private communication, the following analyses of some milk chocolates, most of which were made in America, selected from all the best known makes in that country. (See Table L.)

These results only call for special mention in that, of the better milk chocolates, examined, of which IV. and IX. are typical, the ratio of sugar to milk solids is approximately two to one. In some other cases, the percentage of butter fat, as in V., VI. and XII., is abnormal for the amount of lactose present, if full-cream milk only were used. In the latter case, it is possible that an explanation will be found in the fact that, in America, it is not uncommon to add butter fat, at a later stage, to milk chocolate mass made with



skimmed milk or skimmed milk powder. This practice is adopted for overcoming the "cowy" taste which Americans commonly find in European chocolates. Attention has been called to this question of taste in another place.

With regard to microscopical examination, it is seldom that starchy adulterants are to be found, though the degree of fineness to which the chocolate is ground is noticeably less than in European milk chocolates, sometimes causing a sensation of grittiness or roughness that might be attributed to coarse starch particles.

The commendable efforts of Dubois\* to produce recipes from analyses, when that experimenter was working in the U. S. Department of Agriculture, also show some of the traps into which the chemist, not acquainted with the chocolate industry, may fall. Thus, after making a statement on the estimation of milk solids in milk chocolate by determining lactose, butter fat and casein, he has shown, from analyses of chocolates of known composition, that the calculations of milk solids, based on these estimations, are usually too high. With regard to the estimation of butter fat, Dubois has said: "It was first thought that possibly the Koettstorfer number would assist in estimating butter fat, but it was found that the small amount of butter fat present would not introduce sufficient variation in the number to make it of value in the calculation." Both the Reichert-Meissl and the Koettstorfer numbers of some commercial samples of cacao butter were then shown. A sample of milk chocolate was prepared in the laboratory after the following formula:—

Cacao mass . . . . .	28
Sucrose . . . . .	36
Milk powder (presumably full-cream)	24
Cacao butter . . . . .	12
	<hr/>
	100

\* W. L. Dubois, *U.S. Dept. Agric. Bur. of Chem., Bull.* 152, November, 1911.

Subsequently, analyses of this chocolate and of some commercial milk chocolates were made with the following results :—

TABLE II.—*Analyses of some Commercial and Laboratory Samples of Milk Chocolate (Dubois).*

Kinds of chocolate	Milk solids given.	Lactose.	Fat.		Reichert-Meissl number (fat).	Koettstorfer number (fat).	Butter fat.	Casein.	Milk solids found
			Soxh.	Short.					
	Per cent.	Per cent.	Per cent.	Per cent.			Per cent.	Per cent.	Per cent.
Laboratory.	24	10.6	33.66	30.16	5.5	200	7.0	8.80	26.40
Huyler's	10	3.4	33.02	30.70	2.9	196.4	3.3	2.39	9.10
Peter's	17.4	6.07	33.25	31.00	4.5	199.3	5.5	7.10	18.66
Penna	17.6	6.20	32.42	30.50	4.0	200	4.7	7.20	18.10

In the first column are shown the figures for milk solids, known to have been added in the mixing, supplied, in the three last cases, by the manufacturer. The last column shows the total milk solids, found by analysis. It will be observed that, with the exception of Huyler's chocolate, the figures, obtained by analysis, are higher than those given in the formula, a fact which Dubois was inclined to think was due to the imperfect method of the estimation of casein. Baier \* has provided some analyses of cream chocolates and milk chocolates, of which only the latter are shown in Table LII.

"Ordinary" milk chocolate, like "ordinary" plain chocolate, is the designation under which recipes for inferior brands are described in books dealing with confectionery.

A good example of such a chocolate would result, if prepared from the following recipe :—

\* E. Baier, *Gordian*, 1909, No. 339.

Cacao mass . . . . .	10
Cacao shell powder . . . . .	5
Potato starch . . . . .	5
Sugar . . . . .	40
Vanillin sugar . . . . .	2
Cacao butter or substitute . . . . .	20
Milk powder (skimmed or separated) . . . . .	18
Colouring matter and flavouring . . . . .	To desired tint and flavour.
	<hr/>
	100

The possible combinations of cheap, substitute ingredients are endless, and it is fortunate that the chemist is in a position to detect the addition, especially in such cases as milk and other chocolates which, being compound articles, are without legal standardisation and are, consequently, not studied from this standpoint by those controlling the conviction for fraud under the "Food and Drugs Act."

#### Milk Chocolate Powders.

The remarks made under "Chocolate powders," in the previous chapter, apply here, with the exception that milk powder is added. In Chapter XVIII., Table XLVI., are shown some analyses of milk chocolate powders, which should be compared with the approximate analysis given below.

The milk powder should be full-cream, and no other addition, such as of starch or shell powder, should be permitted.

A good recipe is as follows:—

Cacao mass . . . . .	19
Cane sugar . . . . .	57
Vanillin sugar . . . . .	1
Milk powder (full-cream) . . . . .	23
	<hr/>
	100



TABLE III.—*Analyses of some Milk Chocolates.*

No.	Moisture.		Fat.		Butter fat.		Casein		Proportion of casein to butter fat.		Fat content of the milk.		Total milk solids less butter fat.		Remarks.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	
1	1.94	32.87	8.96	8.12	1:1.10	3.48	31.33	22.37							True milk chocolate.
2	1.84	29.24	8.56	7.89	1:1.10	3.81	30.55	21.90							True milk chocolate.
3	2.69	29.29	4.45	8.82	1:0.50	1.60	29.03	24.58							Not true milk chocolate.
4	2.26	34.48	8.28	7.09	1:1.17	3.68	28.03	19.75							
5	1.68	30.85	7.66	5.32	1:1.44	4.54	22.49	14.83							
6	1.60	35.65	6.60	5.50	1:1.20	3.78	21.93	15.33							True milk chocolate.
7	1.49	34.30	4.32	4.28	1:1.01	3.18	16.25	11.93							
8	1.97	32.24	4.72	4.09	1:1.15	3.62	16.11	11.39							
9	1.32	28.45	6.25	3.45	1:1.81	5.70	15.86	9.61							Not true milk chocolate.*
10	1.00	27.68	1.34	5.12	1:0.25	0.82	15.80	14.26							True milk chocolate.
11	3.59	29.60	4.22	3.80	1:1.11	3.50	14.79	10.57							Prepared by the author with about 15 per cent. milk powder in which the proportion of casein to fat was 1:1.5.
12	1.50	25.42	4.09	4.03	1:1.02	3.20	14.43	10.34							Prepared by the author with full-cream milk powder.
13	1.23	32.03	3.12	3.02	1:1.10	3.25	12.53	9.41							Prepared by the author with condensed milk.
14	1.48	27.12	3.42	2.74	1:1.25	3.93	11.05	7.63							True milk chocolate.*
15	1.59	30.09	3.67	2.62	1:1.40	4.41	10.97	7.30							Not true milk chocolate.*
16	0.86	23.34	1.04	3.25	1:0.32	1.01	10.10	8.06							True milk chocolate.†
17	1.06	32.85	3.74	2.27	1:1.65	5.19	10.07	6.33							True milk chocolate.†
18	1.27	29.40	2.90	2.12	1:1.37	4.31	8.80	5.90							Not true milk chocolate.*
19	0.76	31.45	1.65	2.55	1:0.65	2.05	8.75	7.10							True milk chocolate.†
20	0.93	29.83	2.47	1.58	1:1.53	4.82	6.82	4.40							

\* Chocolate from poor milk.

† These chocolates contain very little milk.

Such a powder will give, on analysis, approximate figures as follow :—

	Per cent.
Moisture . . . . .	1.0
Sucrose . . . . .	58.0
Ether extract . . . . .	15.2
Butter fat . . . . .	6.6
Casein . . . . .	6.0
Lactose . . . . .	7.0

Too often, however, the milk powder will be from skimmed or separated milk, and mixtures of starch and shell powder will be found to be incorporated, with the result that an inferior article will be produced, which will prejudice the public against all such preparations.

#### Nut Chocolates.

Hazel-nuts, almonds, pea-nuts and pine-nuts are all used for producing chocolates of nutty flavour. In many countries and factories, it is customary to include small quantities of nuts, either to produce a characteristic flavour or for softening the hardness of a plain chocolate. The oil, which the nuts contain, lowers the melting point of the cacao butter and can be detected by chemical analysis, as seen in a later chapter.

Special nut chocolates, frequently containing the whole nut of the hazel, almond, pine or even walnut, are to be found in confectionery shops under fancy names which show that such additions have been made. In America, the "Almond Bars" are especially popular, though it is usual to find quite a large proportion of pea-nuts included. Large quantities of nut paste are often added to the cacao mass and sugar in these cases, and, when used, it is certainly desirable that the nuts be roasted previously to being ground and added. Even when the precaution of roasting has been taken, maggots will frequently develop in nut chocolates,

and considerable trouble between manufacturer and retailer may arise from this fact. Besides killing the natural insect life which is present in 90 per cent. of nuts, especially in shelled hazels, roasting enhances the flavour, and it is for this reason also that the process is to be recommended.

Analysis of nut chocolates will always reveal a higher percentage of fat than in the case of plain chocolates, as the nuts, themselves, contain from 30—40 per cent. of oil.

The addition of ground almonds, hazel-nuts, pine-nuts, pea-nuts and walnuts will slightly lower the saponification value of the extracted fat and will considerably raise the iodine value.

Butter fat, or the natural fat of milk, will raise the saponification value by a large amount and will have little or no effect upon the iodine value. The importance and estimation of these values will be considered in later chapters.

A good nut chocolate may be prepared from either of the following recipes :—

	I.	II.
Cacao mass . . . .	28	17
Sugar . . . . .	42	35
Roasted hazel-nuts, almonds or pea- nuts in paste . . . .	27	40
Cacao butter . . . .	8	8
Vanilla . . . . . to flavour.		to flavour.
	<hr/> 100	<hr/> 100

More cacao butter will have to be added if whole nuts are used. The method of procedure for manufacture will be the same as for plain or milk chocolates, already described, though some difficulty may be experienced in moulding, which should be conducted at the lowest possible temperature.

It is unfortunately true that, during the war, milk chocolates and nut chocolates have been subject to adulteration. It is less a question of including harmful ingredients than of supplying to the public a product under a misleading title. We know of cases, where chocolates have been sold under the name of "Milk Chocolates," that only contained minute proportions of milk solids, whilst the remainder of the albuminoids were supplied from treated pea-nuts. Whilst we do not consider such acts criminal, we must term them dishonest.

#### **Date Chocolate, Egg Chocolate, etc.**

To the making of special and fancy chocolates there is no end. Chocolate is one of the few pleasant, yet highly nourishing and comparatively cheap, foods which will blend with most things, even with peppermint, and, as a matter of course, it becomes the plaything of dieteticians and food cranks. Moreover, the well-deserved popularity of milk chocolate, which was the first real departure from the straight article, has raised hopes in the breasts of manufacturers that a similar demand might be created among the masses for such articles as Date Chocolate, Egg Chocolate, Malt Chocolate, etc.; hence the number of patents that can be found for the manufacture and composition of these preparations.

Mapleton's Nut Food Company, Ltd., in a British patent of 1918 (118,052), claims a patent, for preparing food-stuffs, which consists in treating cacao beans so as to produce a paste, reducing fruit from the date tree or date palm to a smooth pulp, and mixing the two together. The proportion of 2 to 3 lbs. of dates to 1 lb. of cacao beans is suggested, and the patent covers the use of apples, pears or apricots for the same purpose. The author has seen this preparation, but has no analysis of it. In 1917, a sample of Date Chocolate from France, however, was analysed and showed the

addition of some 10 per cent. of stoned dates to a normal chocolate. This confection, presumably, had been prepared for supplying a cheap but palatable chocolate during the shortage of cane sugar. The analysis showed :—

*Analysis of a Date Chocolate.*

	Per cent.
Moisture . . . . .	5.56
Ash. . . . .	1.83
Fructose . . . . .	7.40
Cane sugar . . . . .	31.20
Fat . . . . .	28.70
Fat-free cocoa and other solids . . . . .	25.31

An English patent was applied for, in 1914, by Bates for the manufacture and composition of an egg chocolate consisting of a suitable mixture of roasted and shelled cacao, dried milk, dried yolk of egg, agar-agar and sugar.

In 1909 (English Patent 18,337), claim was made for a process for the production of a dietetic and laxative or neutral cocoa or chocolate material, "consisting in adding to the cocoa or chocolate certain micro-organisms or enzymes, being ferments which are able to develop lactic acid in the digestive organs from carbohydrates contained in the cocoa or chocolate without the formation of gases." Numerous instances, similar to the last, could be quoted, but such preparations must have only a very limited sale, so long as the chocolate-consuming public does not seek to find cures in its confections.

## CHAPTER XXI

### MILK CHOCOLATE AND "CHOCOLAT FONDANT"— METHODS OF MANUFACTURE

#### **Milk Chocolate.**

It must be assumed, at the outset, that Milk Chocolate is to be prepared from cacao mass, sugar, full-cream milk and some added cacao butter only, with or without the addition of small quantities of harmless flavourings. This, indeed, is the definition of true milk chocolate, and, apart from the legal aspect, it is impossible to make a pleasant preparation without adhering to this definition. So far as the cacao products and sugar are concerned, they do not call for special mention over and above the qualities already detailed as necessary for good plain chocolate, though the former should be selected for light colour and mild flavour. The addition of the milk, however, is clearly subject to infinite possibilities :— It may be added as fresh milk to the sugar, and the whole boiled down to a consistency suitable to be added to the cacao mass ; it may be added to the sugar or cacao mass as condensed milk, or it may be mixed in with the chocolate mass as a powder. The number of patents on the subject is very large, and each manufacturer, rightly or wrongly, believes that his method of preparation is secret. If the product is good, the manufacturer's pride is not only justifiable but admirable, but too often it is assumed that he is the possessor of a secret which he jealously guards.

The author has been often told that there is only one way of making good milk chocolate, and that is from the fresh milk direct. The fact that the process is laborious does not

necessarily enhance the virtues of the finished product ; on the other hand, the fact that only a few of some of the best manufacturers of milk chocolate have adopted, and have continued to use, the process does not lessen the possibility of accuracy of this whispered secret. Yet, again, the " proof of the pudding is in the eating " thereof, and the author knows of manufacturers who are using this process, but who have not succeeded in convincing him that their product is superior.

Looking at the matter from both the scientific and food specialists' point of view, it is obvious that, if milk is employed, sooner or later the liquid has to be reduced to dry solids in the manufacture of milk chocolate. Secondly, it is clear that, according to the way the milk is reduced to dry solids, so will the chocolate possess a fresh milk-flavour, a pasteurised milk-flavour, a butter-flavour or a rancid fat-flavour. The keeping qualities of the chocolate, too, will depend upon its method of preparation. Therein lies the real secret of the manufacture of milk chocolate, the preparation of which cannot be described in writing, except in its broadest outline, since the final product depends, firstly, on the quality of the milk used, its freshness, its fat-content, etc., and, secondly, on the processes through which it passes, whether it is reduced in an early stage, *in vacuo*, to a concentrated form with or without sugar, or boiled down with sugar in an open pan, or whether it is reduced, direct to the form of a powder, by the " Spray " or " Drum " process. Each possible combination of these processes will give a slightly differing result, till, at the two extremes, milk chocolate prepared from fresh milk, reduced at a low temperature to dry solids, with or without sugar, and that from milk powder, prepared from poor milk at a high temperature, will be as different as cheese from chalk. No writer can do more than indicate the advantages, on the one hand, and the pitfalls, on the other, of any process. Trusting, therefore, in the good sense of the reader,

the author has no hesitation in putting forward the different methods of manufacture in broad outline, knowing full well that the long-established manufacturer of milk chocolate may find the account wearisome and certainly lacking in *minutiae*, to which he may attribute the superiority of his own product.

Milk chocolates are usually preferred light in colour; hence the blend of beans should be determined with this point in view. Both Java and Ceylon cacaos are light brown in colour and mild in flavour, so that, if used, a blend of these cacaos will keep the colour of the chocolate pale and not allow the cacao flavour to predominate too greatly at the expense of the milk. Trinidad, Maracaibo and Venezuelan cacaos may also be used, but their flavour is stronger and their colour is darker than those of Java and Ceylon, and they must, in consequence, be blended according to the requirements of the manufacturer.

In Germany, legislation demanded 12½ per cent. of milk solids in milk chocolates, and it is the general practice, all over the world, for the best milk chocolates to contain at least that quantity, many of the best Swiss makes showing, on analysis, 20 per cent. or more. Again, the best milk chocolates show, on analysis, a percentage of butter fat consistent with the milk solids present, as indicating that full-cream, fresh milk, or condensed milk or milk powder from full-cream milk had been used. Skimmed or separated milk powders are sometimes employed in the cheaper chocolates, with, occasionally, a proportion of full-cream powder, in some of the better brands. It is, however, wrong to suppose that skimmed milk powders are added in order to cheapen the product, firstly, owing to the higher price of such powder over sugar, and, secondly, owing to the fact that a separated milk powder contains practically no fat, and that, consequently, costly cacao butter, or its substitute, has to be added, in order to produce a workable chocolate paste.



With regard to the refinings to which milk chocolates are submitted, it seems desirable here to mention that, though the use of steel refiners is recommended for removing from the paste all grittiness due to cacao or sugar, cold refining on granite rollers is eminently suitable for milk chocolates. This latter process and its advantages have already been described, and, in the following pages, it must be assumed by the reader that it can be conducted after any one refining on the steel refiners, after the chocolate has been block-moulded. The powder, as it comes from the steel refiners, is placed in a "mélangeur" and run with a little added cacao butter; then, after a period in the hot room or hot cupboard, when the temperature should not rise above 50° C., the chocolate is moulded into blocks weighing about 56 lbs. each. The blocks, as soon as cold and well set, are placed on a three- or five-roll granite refiner, which shaves off a thin layer with each revolution of the rollers, and a levigation of the mass is secured that cannot be approached by any other existing system.

The use of the "conche" machine has also already been discussed, and, whilst it is perfectly possible to make good milk or "fondant" chocolate without such a machine, *with* a "conche," results can be obtained which greatly add to the qualities of the finished products. The benefits acquired by the use of a "conche" are several; a very perfect and thorough blending of the ingredients employed, owing to the continuous and often violent motion to which the mass is subjected; a rounding-off of any sharp particles that may have been left over after refining, brought about by the motion of the mass, by friction, and by the heat employed; a smoothness, usually described as "velvety," which is the outcome of thorough incorporation of the ingredients, and of a certain degree of aëration of the mass which is inclined to be flat and "dead" after refining; a development of flavour, secured both by the action of air and heat, depending upon

the temperature employed, which, taking place within the whole mass, is of a more uniform, soft and balanced nature than can be secured by the addition of flavouring matters, caramelised sugar or other ingredients to the mixing.

It should be emphasised, however, that the temperature of the milk chocolate, maintained in the "conche," should not be allowed to rise much above 100° to 110° F., as the milk might acquire an unpleasant flavour, against which it should have been guarded up to the point of incorporation in the mixing. For this reason, prolonged treatment at a moderate temperature in a "conche" is recommended rather than quick "conching" at a high temperature.

*From Fresh Milk.*—The preparation of milk chocolate from fresh milk has formed the subject of many patents. Systems of concentrating the milk with sugar, or with sugar and defatted cacao in mixers by heat, in the open air or *in vacuo*, have been variously devised. In one such system, a mixture of milk and sugar, concentrated in the open air until of a creamy consistency, is added, when still warm, to partially defatted cocoa powder. The mixture, so obtained, is spread out in thin layers and exposed to a temperature of 80° to 100° C., in partial *vacuo*, till dry. Or, again, cacao mass with sugar or milk, or both together, is dried in partial *vacuo* at a suitable temperature.

In making milk chocolate from fresh milk, the milk should be pasteurised as soon as possible, or, if absolutely fresh and warm from the cow, should be reduced in temperature with all possible speed. Concentration may then be undertaken by any of the recognised methods, the object being always to perform the operation at the lowest possible temperature and with the maximum of speed. Hence, *vacuum* condensers are commonly used, and, since sugar has to be employed in the manufacture of the chocolate, it is a more simple operation if the sugar is added to the milk, and both are concentrated together.

The addition of sugar to the milk has a further advantage which is not obvious at first sight. Sugar crystals have to be ground to a fine powder, unless "amorphous sugar" is used, before it is mixed with the cacao mass in the preparation of chocolate. This pulverising of the sugar facilitates intimate mixing and reduces the number of refinings necessary to secure, for the chocolate, a smooth consistency. By mixing it with water and reducing the heavy syrup in the now well-known "amorphous sugar" process, a soft, easily crushed sugar is obtained which greatly assists in the manufacture of "fondant" chocolate. In the presence of invert sugar, or particularly lactose in certain very small proportions, the production of true "amorphous sugar" is greatly facilitated, and, though the same product is not reproduced exactly in the preparation of milk chocolate by concentrating sugar and milk together, crystal cane sugar, in the presence of casein and lactose, is reduced to an "amorphous" state resembling that of true "amorphous sugar," more or less closely according to the care taken in its preparation and concentration. Apart from the refinings on the steel or granite refiners, this largely accounts for the smoothness so characteristic of the best milk chocolates.

A good proportion of fresh milk to sugar, for purposes of concentration, is 100 to 25, which, in the case of a full-cream milk, would provide a proportion of twelve parts of dry milk solids to twenty-five parts of sugar on complete desiccation, the milk containing 9 per cent. of solids other than fat, and 3 per cent. of fat. Frequently, half the total sugar is boiled down with the milk, the remainder being added in the "mélangeur," after the cacao mass, milk and sugar have been dried in the hot chamber.

When the milk has been concentrated sufficiently, according to the requirements of the manufacturer, to a very thick cream or to such concentration that it will just commence to grain on cooling and standing, the sweet concentrated milk is

added to the correct proportion of cocoa powder or cacao mass in a "melangeur" or mixer and thoroughly incorporated. It is clear that there is much water still to be removed. This may be done either by running the doughy mass through a set of granite rollers and drying the thin sheets, so obtained, in a chocolate stove or drying room at about 50° C., or by running the mass down, till partially dry and granular, in a heated "melangeur." The former method is suggested as more suitable when the concentration of the milk has been low, and when there is a very great deal of water to be removed, especially when cocoa powder has been used. The latter method is more suitable when the milk has been condensed to the point of graining, or nearly to that degree, when there will be considerably less water to be removed, and when the violent agitation of the rollers of the "melangeur" will prevent the formation of large crystals of sugar, which would occur if the sweetened milk, at that concentration, were allowed to stand. The milk chocolate paste must be completely deprived of its water, whichever process is adopted, and extra cacao butter has to be added, in order to reduce the paste to a workable condition after the refinings and before moulding.

In the case when the sheets of paste have been dried in the hot room or hot cupboard, it is advisable to pass the mass through a fine sieve, in order to prevent the formation of lumps in the "melangeur" or mixer, when, subsequently, extra cacao butter and remaining sugar (if any) are added. On removal from the "melangeur," the paste is refined and again treated in the hot room for some twelve hours, a heating which removes the remainder of the water and which reduces the paste to a suitable condition for another refining, or for placing in a "conche" machine, at about 45° C., which should be run for a minimum period of forty-eight hours.

When, however, the sweet condensed milk has been added in the highly concentrated state to the cacao mass and

remaining sugar (if any), the "mélangeur" is run until the paste is nearly free from water and has become granular. Then, the chocolate paste or granules must be dried in the hot chamber and, subsequently, refined, with the addition of further cacao butter of which at least half of the total quantity has been kept for this purpose, to secure the required consistency. The operations that follow are further refinings, with further additions of cacao butter, and "conching," until the chocolate has obtained the "velvety" consistency, so much desired. The temperature, 45° C., should not be exceeded, and the "conching" should be continued up to ninety-six hours.

*From Condensed Milk.*—It is more than probable that the chocolate factory will not be situated in a district from which large quantities of fresh milk are obtainable. In such cases, the chocolate manufacturer may establish condensing plants in a good milk district, whence the condensed milk will be forwarded to the factory, or he may purchase condensed milk in the open market.

When the manufacturer, by possessing his own condensing plant, can control the degree and method of concentration of the milk, he can prepare his chocolate in any way to suit his convenience and can manufacture a product similar in every way to that prepared from fresh milk. If, on the other hand, he buys his condensed milk in the open market, he is often faced with the necessity of having to remove some 60—70 per cent. of moisture, which can be done by any of the methods already indicated, though it is probable that he would have to instal a *vacuum* apparatus of some sort, to perform the further concentration in a suitable manner before adding the milk to his chocolate mass. He may, of course, if he wishes, call to his aid the hot room or drying cupboard to assist him in any subsequent drying-out process.

All the subsequent operations will be the same as those already described under the heading "From Fresh Milk."

*From Milk Powders.*—In recent years, the preparation of milk powders has reached a remarkable state of perfection, but it is only in the last few years that the successful production of full-cream milk powder has been accomplished. The difficulties attending the reduction of fresh milk to a dry powder lie chiefly in the fact that high temperatures, even that necessary for pasteurisation, impart to the milk a cooked flavour. For this reason, concentration to condensed form is conducted at as low a temperature as possible in partial *vacuo*. On the other hand, if the milk is not sterilised or the reduction to a powder takes place over too long a period, when oxidation of the fat occurs, or at too high a temperature, when a cooked fat flavour is imparted to the milk powder, the product suffers considerably, and the "cowy" flavour, so often mentioned to the author in America, becomes apparent in the finished chocolate.

Milk may be reduced to a powder, after a preliminary concentration, by passing it in a thin film over a heated drum, or by the "spray" process, when the milk in the form of a fine spray is passed to a heated chamber, which abstracts the water from the fine particles of milk spray, and from which the hot watery vapour is drawn off, leaving the dry powder to fall to the floor. Patents have been taken out for both processes, with numberless modifications both for normal and reduced atmospheric pressures. Again, the object underlying the use of reduced pressure is to lower the temperature to which the milk is heated.

In preparing milk chocolate from milk powder, the latter is added as an ordinary ingredient. The cacao mass, sugar, milk powder and some added cacao butter are thoroughly mixed together in a mixer or "mélangeur." It is advisable to keep out from the first mixing about half the total quantity of cacao butter that will have to be added, since, in the subsequent processes, especially the refinings, the chocolate paste is inclined to dry and to become doughy in con-

sistency. One or more refinings on the steel refiners may be carried out according to the degree of fineness required, and it should be remembered that chocolates prepared with milk powder require more refining than those made from fresh or condensed milk, owing to the granular, slightly hard, particles of milk solids which do not always refine down easily. Treatment in the hot room or cupboards, after each time the paste is run down in a "melangeur" following a refining, is desirable though not essential, and the cacao butter is added continually as the consistency of the chocolate paste demands.

The chocolate comes off the refiners as a fine, dry, light-coloured powder, owing to the fact that the fat has become distributed through the mass which, containing now a greater superficial area to be covered, tends to dry up the liquid fat. It is after these refinings that addition of cacao butter will be found necessary.

As before mentioned, the "cowy" flavour is sometimes overcome in America by using skimmed or separated milk powder which develops less easily the bad effects of high temperature, since it has been freed almost entirely of its fat, and by adding, at later stages in the manufacture of the milk chocolate, fresh, dairy butter fat which, consequently, is not submitted to prolonged or too great heating.

All the processes for the preparation of milk powders are based upon either the Just-Hatmaker (drum process) or the Merrell-Soule (spray) process, the improvements being usually in the method of applying the milk to the drum, the heat employed, or the preliminary degree of concentration of the milk, whilst the use of a partial *vacuum* is yet another modification. The "spray" process stands substantially the same to-day as formerly, with some slight improvements and modifications, and it is certain that this is the most satisfactory method, at present devised, for the preparation of milk powder suitable for the manufacture of milk choco-

late. Owing, however, to the extremely fine state of division to which the milk solids are reduced and to the comparatively low temperature adopted in its preparation, the full-cream milk powder, by this process, is liable to turn rancid quickly on exposure to the air, and precautions to avoid this have, consequently, to be taken.

Through the courtesy of Mr. Edwin P. Carpenter, manager of Casein, Ltd., the author is able to describe the method of manufacture of "Trumilk," which is simply pure, fresh, full-cream milk reduced to powder form.

As soon as the milk is received from the farmers, it is thoroughly cleansed of any impurities that may have got into it through carelessness or inadvertence, by passing it through separators. The next process is pasteurisation of the milk.

The milk is then passed to the *vacuum* pan and reduced to 12 Beaumé, which equals a specific gravity of 1.088. The reason for taking part of the water out of the milk *in vacuo* is that, by so doing, a certain amount may be removed at less cost than by passing it through the hot chambers.

From the vacuum pan, the milk is carried to the various drying chambers or units. Each unit has its separate chamber and pressure pump. The drying chamber is a tiled room about 9 feet square, heated with hot air to a maximum temperature of 76.6° C., into which the milk is sprayed through an aperture about  $\frac{1}{16}$  millimetre in diameter, or about the size of the point of a needle, at 200 atmospheres pressure. This spray is enveloped in a current of hot air, drawn from a battery of steam pipes encased in insulated boxes. An exhaust fan draws filtered cold air through the heated boxes and discharges it into the heated chamber.

The moment the fine particles of spray come into contact with the hot air, the moisture is immediately evaporated and carried off, and the solids of the milk fall to the bottom of the boxes. To prevent loss of any milk solids, the hot air



passes through a series of light cloth filters which detain the powder, but allow the moist air to escape. A shaking motion frees the meshes of the filters from powder which falls into a receptacle below.

The milk powder, so produced, is minutely fine and completely soluble in water, whilst the natural fat, in an aqueous solution of the powder, is in a state of almost perfect emulsion, similar to that of normal, fresh milk. By the above process, the albumen of the milk is retained in its normal form, and the enzymes of the milk are not destroyed.

There is no doubt that milk powder produced by the spray process, correctly carried out, is vastly superior in solubility and flavour to that made by other systems, such as the Just-Hatmaker, which involve the heating of the milk to a minimum temperature of  $100^{\circ}$  C. in order to secure desiccation—a temperature sufficiently high to cook the albumen of the milk, destroy the enzymes and partially caramelize the sugar.

There is no reason why the finest milk chocolate should not be prepared from full-cream milk powder, such as “Trumilk,” which possesses the natural flavour so highly esteemed.

A remarkably uniform product is obtained by the “Trufood” process, which shows, on analysis, the following composition:—

	Per cent.
Moisture . . . . .	1·5
Fat . . . . .	26·6
Milk sugar, etc.. . . .	38·4
Proteids . . . . .	27·6
Mineral matter . . . .	5·9
<hr/>	
Total . . . . .	100·0

Analyses, made by the author, of condensed milk and milk powder, prepared from pure, fresh, full-cream milk, suitable for the manufacture of milk chocolates, are given:—

TABLE LIII.—Analyses of Condensed Milk and Milk Powders.

	Condensed milk.	Full-cream milk powder.	Coutts.*	
			Full-cream milk powder.	Skimmed milk powder.
	Per cent.	Per cent.	Per cent.	Per cent.
Moisture . . . .	64.16	2.50	5.75	7.73
Fat . . . . .	10.92	25.60	26.34	1.35
Proteids . . . .	9.52	27.20	24.40	32.26
Total solids . . .	35.84	97.50	—	—
Ash . . . . .	2.10	5.94	6.55	7.30
Hydrated lactose . .	—	—	36.73	50.38
Cane sugar . . . .	—	Nil.	Nil.	Nil.

The subsequent treatment of the milk chocolate in the "conche" machine is the same as that already described, and it should be continued till the chocolate paste has acquired the necessary "velvety" smoothness, though it is desirable again to emphasise that temperatures above 56° C. should not be employed in the manufacture of milk chocolate.

In moulding milk chocolate, great care has to be taken that the moulds are thoroughly clean and frequently washed. Neglect of this precaution will cause the chocolate to stick to the mould, and dull patches are liable to appear on the surface of the moulded chocolate. The more liquid fats and oils, curiously enough, cause these dull patches on the surface of the tablets, so that plain chocolate, moulded immediately after milk chocolate in the same moulds, will often be found to suffer from this defect, unless the moulds have been thoroughly cleansed after they have been used for the milk chocolate. It is also a fact that milk chocolate can be moulded at a lower temperature than plain chocolate and that the former cools more slowly than the latter. A tempering machine, for obtaining the chocolate at a correct temperature for moulding (about 34° C.), should be employed, if the mass in the "conche" is too cold or too hot.

\* Dr. Coutts, *Local Government Board Food Report*, 24, iii., 1918.

**“Fondant” Chocolate.**

As has already been stated, “fondant” chocolate differs from ordinary plain chocolate only in its “velvety” smoothness, secured by an increased percentage of added cacao butter and by the extremely fine state of division to which each particle of the chocolate mass is reduced. Analyses of “fondant” chocolates have been given in a previous chapter, and it will be realised that it is largely the method of preparation that secures for these chocolates the individuality characteristic of many well-known brands. On the other hand, care in selection of the blend of beans must not be ignored, and the characteristics of the different cacaos must be borne in mind when constructing a recipe.

A “fondant” chocolate may be mixed from the following extremes of cacao mass and sugar, the higher the percentage of sugar, the more cacao butter that, necessarily, must be added :—

Cacao mass	.	.	.	25 to 45 parts.
Sugar	.	.	.	60 to 50 „
Cacao butter	.	.	.	14 to 4 „
Vanilla or Vanilla sugar	.	.	.	1 „
				<hr/>
				100 to 100 „

The cacao mass should consist of partially roasted nibs of a good blend of cacaos, should be free from husk, and milled to a fine state of division. The sugar should be either finely pulverised crystal sugar or, better, “amorphous sugar,” prepared according to the method briefly outlined in a former chapter. All the cacao butter should not be added to the mixing at the beginning, but at least half should be kept back to add, a little at a time, to the chocolate mass, placed in the “melangeur” after each refining.

Subsequent processes are substantially the same as in the manufacture of milk chocolate. Thorough mixing followed

by refining on the steel refiners, a short working in the "mélangeur" with a little added cacao butter, a short treatment in the hot room, blocking off and cold refining on granite rolls, further treatment in the "mélangeur," a second refining on the steel refiners and another cycle or two of these processes, as desired, are all identical with the procedure adopted in the manufacture of milk chocolate. It is in the last stage, when the chocolate is heated and worked in the "conche" machine, that the procedure differs. If the cacao nibs have been fully roasted, the processes of manufacture of "fondant" chocolate may be said to be identical with that for milk chocolate, since the temperature should not be allowed to rise sufficiently to cause a further roasting of the cacao. In mixing, say for thirty minutes, the mass does not rise above 45° C., and each refining on water-cooled, steel rollers lowers the temperature of the mass to about 30° C. Each treatment in the "mélangeur" for an hour or so does not again raise the temperature above 45° C., and the hot room should not be above 60° C. Thus, in normal working for fully roasted beans, the maximum temperature, reached, is about 50° C. If the chocolate mass is then placed in a "conche" machine, the temperature should be maintained around the maximum employed up to that time, if consistency alone is sought, though, in order to impart the slight caramel flavour also, a temperature not above 65° C. should be employed, as, otherwise, the cacao will burn or become over-roasted.

With partially roasted cacao, however, the final roasting and development of flavour are allowed to take place in the "conche" machine, and higher temperatures and longer treatment are, therefore, necessary. The general rules for roasting cacao beans for a half-roast have already been given in an earlier chapter, and it is clearly impossible to state exactly how long, or how much, the chocolate mass should be subsequently treated in the "conche," in order that the full

flavour of the cacao should be developed. But, it may be stated broadly that a gently applied heat over a long period gives the best result in a "conche" for "fondant" chocolate, not only because the finishing point can be more easily determined, but also because the developing flavour is more evenly attained and distributed through the whole mass.

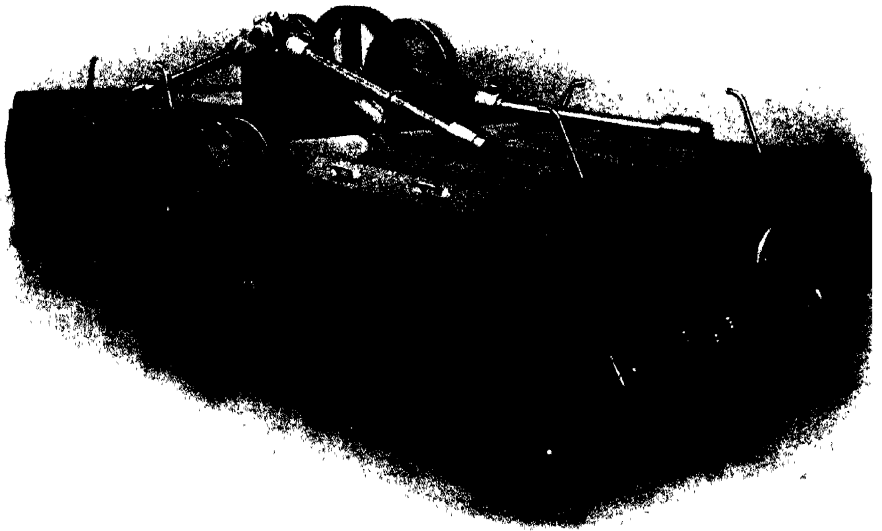


FIG. 24.—Large Size, New Design "Conche" Machine. (See p. 322.)

*By permission of Messrs. Joseph Baker, Sons, and Perkins, Ltd.*

### The "Conche" Machine.

The modern "conche," or horizontal machine, has been designed for the purpose of giving a fine finish in appearance, flavour and consistency to milk and "fondant" chocolates. It consists, essentially, of a tank with granite, or steel, bed upon which are made to run, to-and-fro, granite rollers

attached to a long arm, fixed to one side of a central circular drive.

The tanks are capable of being heated to high temperatures, for the purpose of carrying out the Swiss half-roast process of manufacture, and are often constructed of large capacity and in batteries. In the machine, illustrated, (Fig. 24) there are four tanks, together capable of holding from 2 to  $2\frac{1}{2}$  tons of chocolate, and the floor space occupied is, approximately,  $18\frac{1}{2}$  feet by  $7\frac{3}{4}$  feet. The special advantages of the "conche" have already been considered in Chapter XVI.

## CHAPTER XXII

### COVERING-CHOCOLATE — MANUFACTURE AND ANALYSES — USE OF "CHAMPION" COATING MACHINE, AND "ENROBER."

IN Chapter XVI., the quick methods of manufacturing covering-chocolate have been discussed, and the value of the "Bausman Disc" machine, "Walker" machine and combined steam-jacketed kettle and "melangeur," etc., has been considered.

In the manufacture of covering-chocolate, no less care should be expended, if a good preparation is to result, than should be taken in the manufacture of plain eating-chocolate. It is often imagined that any chocolate is good enough for covering soft centres, fruits, biscuits, etc., and it is not uncommon to find the outside coating of these confections to be little better than powdered husk, sugar and cacao butter substitutes, artificially flavoured. This is a very great mistake, for, whilst it is true that the state of refinement need not be so intense, owing to the gritty nature of the centres, a poor-flavoured chocolate greatly detracts from the subtle blending of flavour that should be experienced from a correct combination of fruit or other confection with that of cacao. There is nothing so pleasant as a cherry cream flavoured with Kirsch, the whole covered with a thin coating of fine chocolate, or the delicate flavour of orange-flower water in combination with almonds in a good, soft nougat, that blends perfectly with a good covering of chocolate, on the palate. But, on the other hand, a rough covering-chocolate, strongly flavoured with coumarin, so often encountered at the present time, is positively objectionable in combination with even the finest flavoured centres.

The method of manufacturing covering-chocolate is, up to the point of refining, identical with that of the best eating-chocolate. The best beans are selected, carefully roasted and shelled, and the nibs milled and mixed with sugar and a sufficiency of cacao butter to bring the whole into a plastic mass. Subsequent treatment depends upon the system of refining to be employed. Since, in order to secure an even and not too thick a coating, the chocolate must be very limpid, there must always be in covering-chocolate a larger percentage of fat than will be found in the best eating-chocolates. This certainly simplifies the manufacture of covering-chocolate.

The price of cacao butter does, of course, to a large extent govern the amount of fat that will be found in covering-chocolates. Thus, in 1914, analyses, made in the author's laboratory, showed 38—40 per cent. of fat as the usual amount present in the best known coverings, whilst more recently, in 1920, covering-chocolates, purchased in the open market, showed figures more near to 35—37 per cent. Chocolate containing anything above 35 per cent. of fat will work well for covering centres, such as creams, biscuits, etc., though the higher figures, 38—40 per cent., would be preferable for machine covering, and in cases where only a very thin coating of chocolate is required. For chocolates going to warm climates, the amount of fat should be kept down to a *minimum*, as a very soft chocolate is inclined to run and to discolour easily.

A movement began, in 1908, at the Geneva Conference, to standardise chocolate, but there was, very naturally, a great divergence of views as to the desirability of such a standardisation, for the most obvious of reasons that each manufacturer had developed his own system of manufacture and was accustomed to include any ingredients that he thought fit, in accordance with his conscience. One British firm suggested that covering-chocolate should be defined as that



"which contained additional cacao butter or wholesome vegetable fat, the total fat amounting to 31 to 32 per cent." N. P. Booth, analytical chemist to Messrs. Cadbury Bros., pursued the subject again at the "Seventh International Congress of Applied Chemistry" in 1909, when he deplored the fact that, in Great Britain, any compound containing but a small percentage of cocoa could be sold under the name of chocolate. Booth suggested definitions, amongst them being one for chocolate-covered goods as "denoting forms of confectionery covered with chocolate, the composition of the latter agreeing with the definition of sweetened chocolate." Sweetened chocolate, he suggested, should be defined as "A preparation consisting exclusively of the products of roasted, shelled, finely ground cocoa beans, and not more than 65 per cent. of sugar, with or without a small quantity of harmless flavouring matter which, if present, should not exceed 2 per cent."

As it stands at present, covering-chocolate may consist of anything tasting similar to chocolate. That this was true then, as now, is seen by the writings of one author of pre-war days, who states that the idea of standardisation of chocolate was looked upon with favour by the makers of the more expensive chocolates, but pointed out that "it would exclude a large multitude of articles now sold as chocolate, and which, while wholesome and toothsome, might be placed at a disadvantage if too limited a definition came to be adopted." There was determined opposition on the part of those who carried on what was regarded as a "perfectly legitimate and acceptable trade."

At the "International Congress of Cocoa and Chocolate Manufacturers" held in August, 1911, at Geneva, resolutions were passed defining Cacao paste, Cocoa powder, Chocolate paste, Chocolate powder, Milk chocolates and Covering-chocolate on the lines adopted at the Geneva Congress of 1908, but the Secretary of the "British Manufacturing

Confectioners' Alliance" frankly stated that the important manufacturers, whom he represented, would not, as a whole, recognise any resolutions which the Congress passed as in any way binding upon the British industry. Some British manufacturers, indeed, had shown their determination to keep their hands entirely free, by refusing to take any part whatever in the proceedings, even to the extent of giving him their proxies.

A good covering-chocolate may be prepared from the following recipe :—

Bitter chocolate . . . . .	30
Sugar . . . . .	49
Vanillin sugar . . . . .	1
Cacao butter . . . . .	20
	<hr/> 100

It should be understood, however, that the manufacturer, who considers the quality of his products, cannot afford to use inferior cacao mass for his covering-chocolate. Just the same care, as is exercised in selecting and treating the beans for eating-chocolate, should be taken in the preparation of covering-chocolate, and all the other ingredients should be of the best.

The next table shows some analyses of covering-choco-

TABLE LIV.—*Analyses of some Covering-Chocolates.*

	I.	II.	III.	IV.	V.	VI.	VII.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Moisture . . . . .	1.00	0.84	2.34	1.12	2.10	0.90	2.50
Ash . . . . .	1.26	1.14	1.60	1.20	1.90	1.42	—
Total fat . . . . .	42.18	35.28*	36.30	34.41	33.78*	35.10	34.78
Cane sugar . . . . .	45.09	50.82	44.92	49.50	50.90	51.03	51.00
Dry fat-free cocoa and added starch, if any .	10.46	11.92	14.84	13.71	11.32	11.95*	10.51
Added starch . . . . .	Nil.	Trace.	Nil.	Nil.	Some arrow-root.	Nil.	Nil.

\* Cacao butter substitutes detected.

lates, made in the author's laboratory, at different times. Considerable variations occur, but the analyses have been made sometimes from bulk samples of chocolate, sometimes from the chocolate removed from biscuits, soft cream centres, etc., and the latter are, therefore, subject to such imperfections as may arise from the technical treatment of the

TABLE LV.—*Analyses of some American Plain and Milk Covering-Chocolates.*

	I.	II.	III.	IV.	V.	VI.	VII.
	Plain	Cocoa liquor.	Milk coating.	Milk coating	Plain.	Milk.	Milk
Cane sugar . . . . .	45.00	N. /	31.97	48.13	48.35	44.76	56.24
Ether extract . . . . .	36.05	49.95	35.55	33.96	34.19	35.20	32.75
Crude fibre . . . . .	1.34	3.07	—	—	1.30	—	—
Ash . . . . .	1.53	3.93	—	—	1.48	—	—
Water-insoluble ash . . . . .	0.89	2.11	—	—	—	—	—
Water-soluble ash . . . . .	0.64	1.82	—	—	—	—	—
Acid-insoluble ash . . . . .	0.16	0.14	—	—	—	—	—
Alkali-insoluble ash . . . . .	1.2	2.15	—	—	—	—	—
Alkali-soluble ash . . . . .	0.6	1.60	—	—	—	—	—
Reichert-Messl. value of fat . . . . .	0.45	0.30	5.76	3.41	1.28	3.85	1.55
Saponification value of fat . . . . .	196.4	194.9	—	—	200.0	—	—
Iodine value of fat . . . . .	36.1	37.4	—	—	35.2	—	—
Refractive index at 40° C. . . . .	—	—	—	—	1.4567	—	—
Crude fibre on sugar-fat-free basis . . . . .	7.07	6.13	—	—	—	—	—
Ash on sugar-fat-free basis . . . . .	8.08	7.85	—	—	—	—	—
Water-insoluble ash on sugar-fat-free basis . . . . .	4.70	4.22	—	—	—	—	—
Water-soluble ash on sugar-fat-free basis . . . . .	3.38	3.63	—	—	—	—	—
Alkali-insoluble ash on sugar-fat-free basis . . . . .	6.30	4.30	—	—	—	—	—
Alkali-soluble ash on sugar-fat-free basis . . . . .	3.10	3.20	—	—	—	—	—
Acid-insoluble ash on sugar-fat-free basis . . . . .	0.84	0.28	—	—	—	—	—
Butter fat . . . . .	—	—	7.96	4.28	—	5.00	1.46
Casein . . . . .	—	—	—	—	—	4.05	1.50
Lactose . . . . .	—	—	7.80	4.70	—	6.05	2.21
Milk solids . . . . .	—	—	—	—	—	15.60	5.47
Microscopic examination . . . . .	—	—	No adulterant.	No adulterant.	No adulterant.	—	Much corn starch.

chocolates during covering and from the centres which they cover.

The above figures, sent by Dubois to the author, show the analyses of some American plain and milk covering-chocolates (Table I.V.).

It will be seen that both plain and milk chocolate coverings are made, the latter particularly in America, where the public seem to like very light milk coverings and very dark plain chocolate coverings, so much so that, in the latter case, artificial colours are often added to produce dark or almost black chocolates.

The analyses, shown, do not call for any special remarks, the chocolates, both in England and America, being very similar in composition. On the other hand, so many of the chocolate coverings, examined, have been found to contain cacao butter substitutes that, since it is known also that this method of cheapening the goods is commonly practised, it is high time that some form of legislation existed on the subject. The practice of adding cacao butter substitutes is harmless in itself, seeing that the fats, used, are quite innocuous and equal to cacao butter in food-value, yet it is certainly desirable that the public should know what it is eating, and, for this reason, that the addition of cacao butter substitutes to any form of chocolate should be notified on the label of the container holding the confections.

The addition of "substitutes" for cacao butter to covering-chocolate may have the advantage of raising the melting point of the combined fats, though palm oil, recommended by more than one confectioner for the purpose, has not this advantage, seeing that its melting point is slightly lower than that of cacao butter. Many of the vegetable stearins can, however, be used to raise the melting point of the chocolate and, as before remarked, though without any deleterious effect upon the health, their presence should be notified on the label for the public benefit.

**Tempering Machinery.**

In preparing covering-chocolate for the covering tables or covering machines, it is necessary to ensure that the

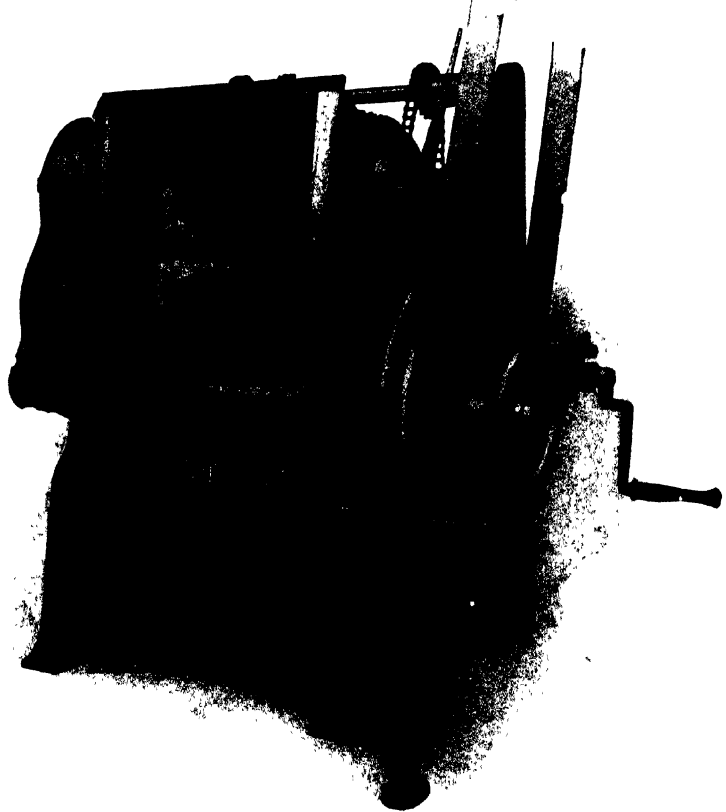


FIG. 25.—Steam-jacketed Chocolate Mixing and Kneading Machine. (See p. 331.)

*By permission of Messrs. Joseph Baker, Sons, and Perkins, Ltd.*

chocolate paste is at the correct temperature. For this purpose, tempering machines, such as have already been described for obtaining chocolate at the proper temperature for moulding, can be employed. A water-jacketed "melan-

geur," or steam-kettle, may be used, or a chocolate kneading machine, as illustrated (Fig. 25), will be found very convenient and suitable. This machine has a steam-jacket, for warming the chocolate, and is provided with two speeds and a reverse motion which is used for discharging. The mixing arms have been specially designed to ensure uniform kneading throughout the mass. Machines to contain as much as 1,800 lbs. have been built, and the chocolate manufacturer can rest assured that there is no more useful an addition to his factory than one or more of these mixers or kneaders, which can be put to a number of uses besides that of tempering or kneading chocolate for covering purposes. The writer has seen this machine used for making the original mixing instead of a "melangeur," for blending alkali with the nibs, and for subsequently drying out the moisture, for all of which purposes this mixer, blender or kneader is eminently suited.

#### **Process of Covering "Cream"-Centres, Biscuits, etc., by Hand and by Machine.**

The application of chocolate to the exterior of sweet confections, candies, biscuits, etc., has received a great deal of attention on the part of the manufacturer. Many chocolate-covered goods are "things of beauty" if not "joys for ever," and their attractiveness in appearance goes far towards establishing a demand.

In small chocolate factories and for the highest class of fancy chocolates, all the covering is performed by hand, not only because a machine would be out of place in dealing with comparatively small quantities, but because the softness of consistency of many of the best centres necessitates the most delicate handling.

*Hand-covering.*—Hand-covering is usually conducted by girls who sit round a metal table, kept at the required

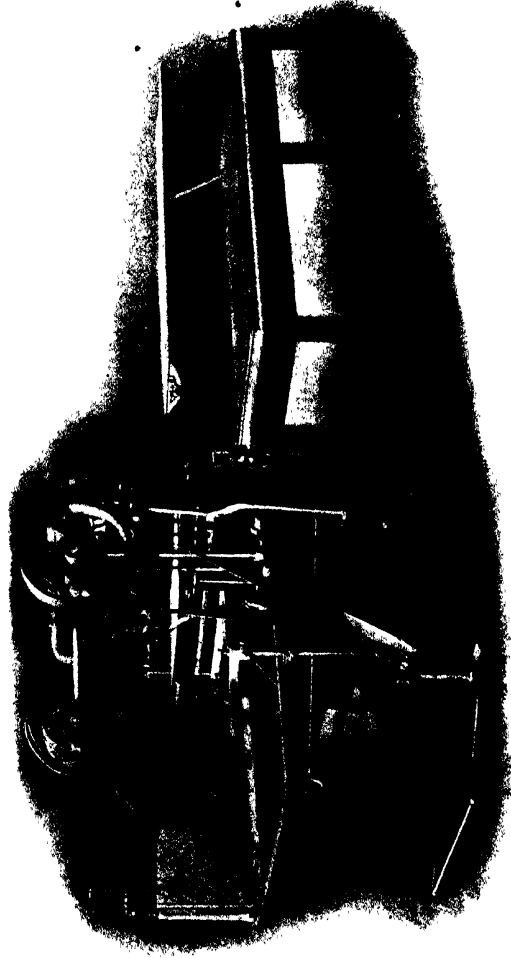


FIG. 26.—“Champion” Coating Machine. (See p. 333.)  
By permission of Messrs. Joseph Baker, Sons, and Perkins, Ltd.

temperature, upon which are placed small quantities of the liquid covering-chocolate. The chocolate must be about  $31^{\circ}$  C. when used for covering, and the finish of the goods

depends very greatly upon the steady maintenance of temperature.

In many factories, the centres are dipped, literally, by hand, that is to say, the cream, nougat, etc., is dipped with the fingers into the chocolate, transferred to a highly glazed piece of paper or "plaque," and the decoration put upon it with the tip of the finger. In other factories, a light two-pronged fork is used, and the "cream" is dipped beneath the chocolate in a bowl in front of the operator, who puts the marking upon the chocolate with the prongs of the fork.

As the correct temperature for cooling chocolate to obtain the best finish has already been discussed, it is not proposed here to deal with the various methods of cooling. Suffice to say that many of the chocolates appearing on the market are spoiled by inattention to this consideration.

We will now turn to two methods of covering by machine.

*The "Champion" Coating Machine.*—This machine (Fig. 26), manufactured by Messrs. Joseph Baker, Sons, and Perkins, Ltd., is designed for the coating of basket goods in large quantities.

The machine consists of a double cast-iron pan, formed to make a well for the chocolate, and an inclined table, to drain the chocolate from a tapping table back to the well. The pan is heated by means of a hot-water chamber between the inner and outer pan, and the chocolate is kept at a uniform temperature in the well by means of a rocking agitator, which is run independently of the dipping and tapping.

The goods, to be covered, are held in position in the baskets, during dipping, by means of a wire screen, suspended over the basket, which is released on the down motion of the basket but which is held firmly on the top until the basket has returned to its stationary position. The screen is then automatically lifted, allowing the basket to be moved forward on runners to the tapping brackets, and another



basket is placed in position from behind the machine, ready for the dipping operation when the clutch is released.

The dipping movement is controlled by means of a lever which releases a clutch, and, on completion of one dipping motion and the return to the top of the stroke, the clutch is thrown out automatically, and the dipping arms remain at rest.

The tapping guides are supported in slides by two vertical rods, attached to a cross-lever controlled by the pressure of the operator's foot, so that light or heavy tapping can be obtained at will.

When sufficient tapping has been applied, a sheet of glazed paper, or a tin "plaque," the same size as the basket, is placed on the top and held in position by means of a light hand-board. The supporting arms holding the basket are tipped over on their axles, till the basket is inverted, when the covered goods drop on to the paper or tin and are ready for the cooling room.

It should be observed that the baskets are, in reality, wire trays, divided into partitions of sizes suitable for the individual goods, to be covered. Girls fill the trays or baskets with the centres to be covered by hand, taking the empty basket from the side supports of the steam-heated drip-tank into which the emptied trays have been slid, after removing the contents on to the paper or "plaque." It is advisable to place the trays or baskets, during filling, on steam-heated metal-covered tables, so that the chocolate does not solidify on the wires.

Six baskets or trays are supplied with each machine and are, of course, made to suit the customer's goods.

Another great point in favour of this machine is that it is self-contained on its frame and requires no foundation.

We have seen the 2-h.-p. "Champion" Dipper at work in many places, and the machine is especially suitable for

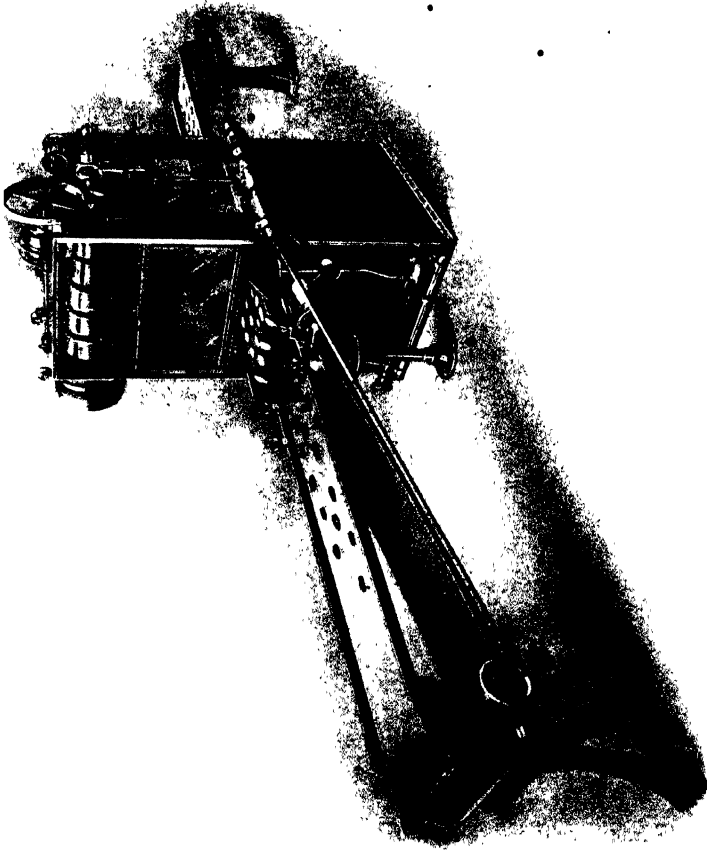


FIG. 27.—The "Enrober" Covering Machine. (See p. 336.)

covering biscuits and medium-sized goods which usually take a very large quantity of chocolate to cover. For this reason, the "Champion" Coating Machines, with the violent tapping action are to be preferred for this class of work to the "Enrober," which requires a more liquid chocolate for

satisfactory working, or a higher temperature, which is apt to impart a reddish colour to the chocolate on the finished goods. Any amount, from 10 to 20 cwt., of chocolate pieces, according to the size of the goods to be covered and to the skill of the operator, can be turned out by one of these machines *per* day, and three girls, only, are required to attend each Dipper.

*The "Enrober" (Fig. 27).* (*Messrs. Savy, Jeanjean & Cie., Paris.*)—Although made up of a great many parts, the "Enrober" is constructed on a very simple principle. It consists, essentially, of an iron case, about 3 feet square and 7 feet deep, which carries the chocolate tanks, hoppers, heating apparatus, belt-conveyers and driving gear. It will turn out anything between 2,000 and 6,000 lbs. of finished goods in ten hours.

The pieces are fed into one end of the machine on a travelling canvas belt; they then pass on to a wire belt-conveyer, within the case, where they travel through a cascade of liquid chocolate. The surplus chocolate is removed by an air blast and by a slight tapping movement, and the goods, after leaving the case, pass on to "plaques" on another travelling web-belt, whence they can be removed to a cool room or passed continuously through the cooling chamber, attached to the machine.

Except when placed on the feeding belt, the goods never need to be touched by hand, until ready for packing. An automatic feeder has now been devised for distributing the goods on the feeding belt, and one girl, with this attachment, can keep the "Enrober" busy.

There are two or three refinements, more recently made to the "Enrober," one of which is the "bottoming" attachment which allows a specially heavily coated bottom of chocolate to be put upon the goods. Another refinement is the Kihlgren system for decorating the tops of the chocolates. By the Kihlgren system, the goods emerge from the "Enrober"

and pass beneath minute streams of chocolate, squirted through very narrow pipes, the speed and direction of the attachment determining the pattern.

Only 3 h-p. are required for driving the "Enrober," including the Kihlgren and "bottoming" attachments, the "Enrober," alone, only requiring 2 h-p. With the Kihlgren attachment, the output falls by about one-seventh, as the goods have to be adjusted, to a nicety, as they pass beneath the streams of chocolate.

Though this machine is, in every way, satisfactory for coating creams, nougats, biscuits and even larger articles, it suffers from one disadvantage, namely, that the chocolate must be in a very liquid state, if the coating of chocolate is not to be thick. This implies either that a large quantity of cacao butter must be added to the covering-chocolate or else higher temperatures, that are inclined to leave the articles with a poor finish, must be employed. The same remarks apply, at present, to all automatic covering machines.

The working process is as follows :—

The chocolate is first put, at a temperature of about 90° F., into the chocolate tank under the machine, the temperature of which is regulated by the steam-heated, water-jacketed tank, so that the bulk of chocolate is maintained constantly at the required temperature. The chocolate is continually stirred in this tank by means of revolving paddles.

The goods are fed on to the first feeding table between steel rods which ensure that they do not touch each other. Travelling along this table on a moving canvas band, they are delivered on to a short revolving wire which, in turn, carries them over the roller of the bottoming attachment. This attachment consists simply of a small water-jacketed tank containing chocolate in which the bottom half of this roller is immersed. In passing over this roller, the goods, in

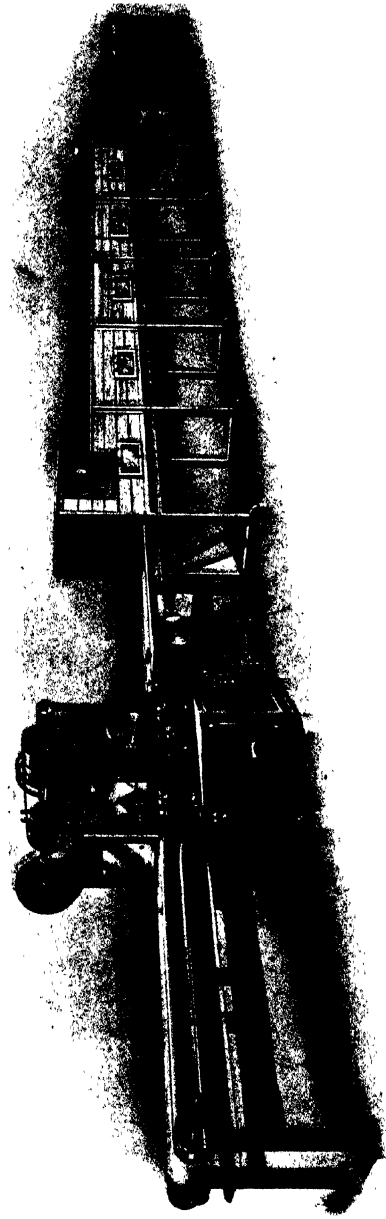


FIG. 28.—The "Enpholda" Automatic Covering Machine, with Cooling System. (See p. 340.)  
*By permission of Messrs Bramah & Co., London.*

the first instance, are covered on the bottom with chocolate and delivered on to another canvas band which runs on top of a shallow tank, filled with cold water. By the time the goods reach the end of this cooling table, the chocolate on the bottom is set, and the pieces are delivered on to a long travelling wire band passing through the machine proper; here, the chocolate, in the tank in the bottom of the machine, is lifted by a revolving drum elevator and delivered into a distributing box, from which it is poured in two streams over the goods as they pass through on the wire band, the surplus chocolate running down through the wire and returning to the main tank in the bottom of the machine.

Immediately following the distributing box, the goods, now covered with a thick coating of chocolate, pass underneath the outlet from a fan, and the current of air, so produced, blows off some of the surplus chocolate. Following this, the band is agitated by ratchet pawls, by means of which the goods are shaken, so as to distribute the remaining covering of chocolate smoothly and evenly over the whole surface.

This operation being completed, and the goods having reached the end of the wire band, the pieces are now delivered on to the delivery table which carries the embossed paper "plaques" on a travelling canvas web, the goods being placed in straight lines on to these papers as they go round.

The outer end of this delivery table passes into the cooling chamber, enough space being left between the machine and the entrance to allow operators to perform a certain amount of hand-marking on the top of the pieces, when required.

The speeds of all travelling bands, chocolate elevator and fan are capable of being varied, independently, according to the class of goods or thickness of covering, required, and all the controls are brought to within easy reach of the senior operator of the machine.

*The "Enpholda" (Fig. 28). (Messrs. Lehmann, Dresden.)*  
—This machine has been specially designed for simplification in construction, so that all the working parts can be readily reached. In other respects, it resembles the "Enrober" very closely. The makers of the "Enpholda" claim several advantages over other machines, used for the same purpose. For instance, the tank containing the covering-chocolate runs on wheels and can be drawn out, thus making it conveniently accessible.

This tank has, too, a large outlet and will automatically empty itself, when it is desired to change any of the covering-chocolate. The travelling wire band, unlike those on other covering machines, is made in sections, so that not only is risk of sagging overcome, but, in cases of breakage, the part can be more easily replaced, and, if spare bands are not available, the remaining bands can be continued in use. The heating of the working parts is done partly by steam and partly by electricity, or gas. The chocolate mass is, moreover, brought up by a pair of rollers which grind any small lumps that may occur in the mass, thus removing all risk of clogging.

The centres pass beneath the cascade of chocolate in the same way as in the "Enrober," and, by a mechanical arrangement, the chocolate is also raised from below, thus covering the bottoms as well as the tops of the goods, while they are travelling on the endless wire band. A blast of air from a fan, as well as a shaking apparatus, removes the surplus chocolate, and, by adjustment of these devices, almost any thickness of covering can be secured. On leaving the covering machine, the covered goods pass continuously through the cooling chamber and are delivered at the other end cooled, finished and ready for packing.

## CHAPTER XXIII

### AROMA AND FLAVOUR DUE TO VARIETY OF BEAN, PROCESS OF MANUFACTURE, AND FLAVOURING MATERIALS, SUCH AS VANILLA, SPICES, ESSENCES, ETC.

THE production of individual flavours in cocoas and chocolates is attained by : (1) selection of special variety or blend of cacao beans ; (2) special process of manufacture ; (3) addition of flavouring materials, such as vanilla, spices or essences.

#### **Variety of Bean.**

In Chapter VIII., the principal characteristics of the different varieties of cacao beans have been dealt with, but the reader must have experienced some difficulty in imagining, as the author had in detailing, the various flavours which, though characteristic, are almost impossible of written description.

Venezuela, Ecuador, Guayaquil, Ceylon, certain West Indian and African beans may be said to be typical of the cacaos for preparing chocolates of the highest class, but, even with these varieties, blending is desirable, as less variation will occur in the flavour of a mixture of beans than in the individual kinds.

Caracas beans are pronounced and characteristic in flavour, Guayaquil Arriba slightly scented, Trinidad bitter and rather strong, and Surinam similar to Caracas. All such cacaos will require blending with those of less pronounced flavour, if chocolate of fine quality is to be produced.

Those beans of more bitter and harsh taste, such as certain of the Ecuador, Para, Bahia, Guiana and Trinidad



cacaos, will require toning up with beans of higher quality, whilst the cacaos of insipid or neutral flavour, such as those of Haiti and St. Domingo, will be improved by blending with beans of more pronounced character.

Further discussion on this subject is useless, for experiment with the different beans will be of more use to the manufacturer, who may require a certain flavour, than any description that it is possible to give.

Improvement in flavour may be brought about in beans of musty odour or great bitterness by treating them with water or alkali solution on arrival at the factory. The processes, which have already been described in Chapters X. and XIII., consist of a post-fermentation, in the former case, and a modification of the bitter and astringent bodies in the beans by treatment with alkali, in the latter. Besides improvement in flavour, it is claimed that these operations give better colour to the kernel and, consequently, to the chocolate, prepared from them. Many of the lower grades of cacao, that come into the market covered with mould, and of a pale interior, may be treated in this way, with advantage.

#### **Process of Manufacture.**

Treatment with alkali, temperature of roast, degree of fineness to which the chocolate is ground, period of working in the "melangeur" and "conche," storage and packing of the finished chocolate, all play an important part in determining flavour.

It has already been pointed out that treatment with alkali is supposed, by some, to enhance the flavour of a cocoa preparation, but, whilst this may be so with a cocoa powder which has the additional advantage of being rendered "soluble," it is not our experience that improvement is to be found in a chocolate, so treated.

The presence of the small quantities of soda or potash, which the treated chocolates contain, is quite harmless,

though there exists a certain prejudice against the use of chemicals in the manufacture of foodstuffs.\* On the other hand, it is well known that even a small quantity of alkali in the presence of fat, especially when heat is applied, must produce a small quantity of soap or saponified fat. This has, however, already been discussed and has been shown to be open to dispute. Saponification of a fat, in all cases, seems to develop the pungent and unpleasant portion of the flavouring matter of that fat, and, though it can be found stated by more than one authority that the addition of alkali prevents a fat from turning rancid, we are convinced that, unless very carefully and judiciously applied, the alkali will produce compounds of fat far more unpleasant to the palate than fatty acids from rancid fat, present in the same proportion. Since, however, it can be proved that the addition of alkali neutralises the tannic acid and other acid astringent matters, and, if the production of saponified fat is insignificant, it must be admitted that a certain amount of good, apart from "solubility," has resulted by the treatment, for the bitterness and astringency of certain manufacturers' chocolates are serious hindrances to the full appreciation of their flavour.

The temperature of the roast determines whether the aroma of the beans has been fully, under- or over-developed. When the cacao is fully roasted, the beans have lost the acid, vinegary smell, and the nibs are free from any objectionable or musty odour which they may have acquired during the processes of rendering them fit for market. Moreover, they have developed the pleasant aroma typical of chocolate, which can best be judged away from the room in which the roast is taking place, preferably in the open air.

Under-roasted beans are most unpalatable, when made up into chocolate, and can best be described by the word "green," signifying a flavour of raw vegetables.

\* *Vide* discussion in Reports of Congress at Geneva and Paris, 1908, 1909, and Chapter XXIV.

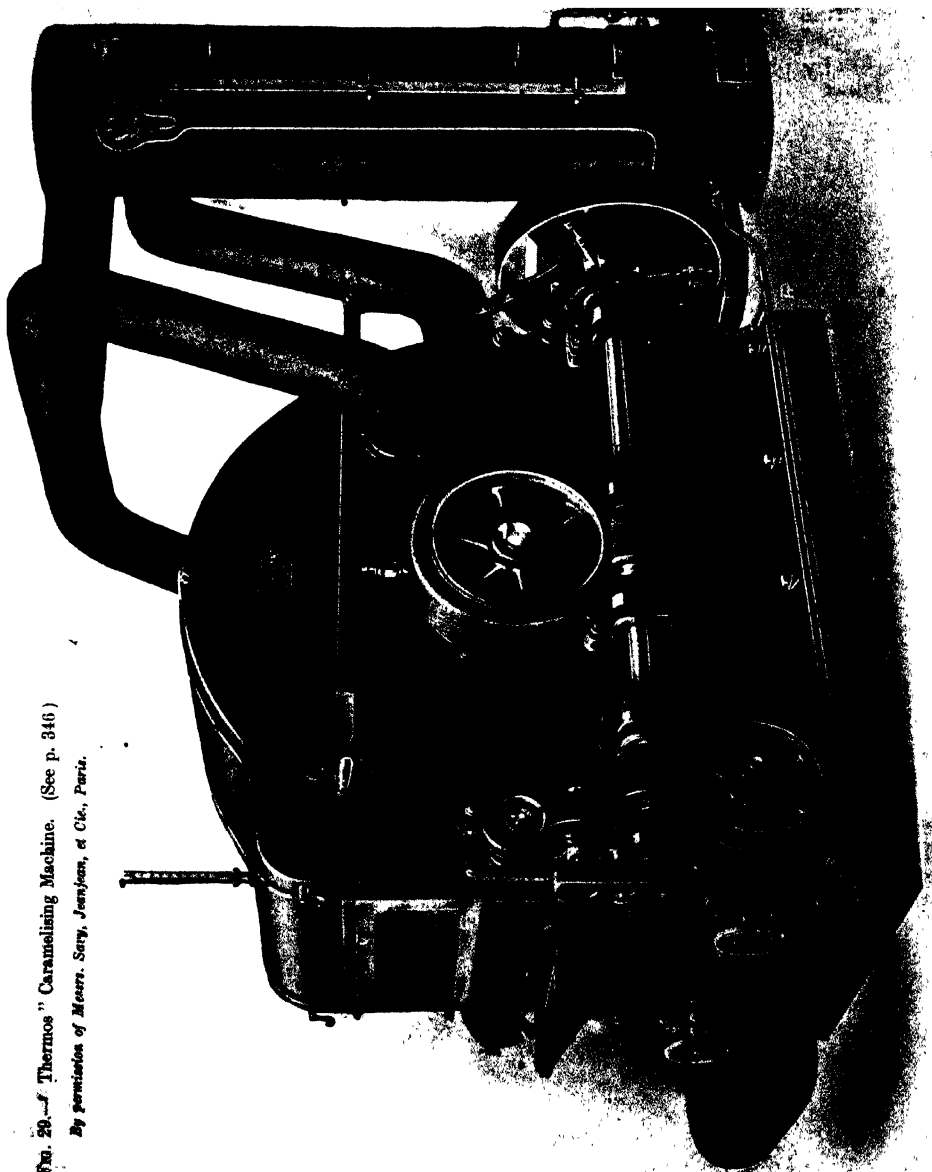
Over-roasting, if unaccompanied by a smoky flavour, ... not altogether unpleasant, if not excessive, and there is little doubt that the native and original Mexicans, who practised the art of chocolate making, roasted their beans to a higher degree than is now customary. The burnt flavour of over-roasted beans is considered, by some, to be pleasant to the palate, and certain "Mexican" chocolates of commerce are prepared from beans that have been severely roasted or by the addition of small quantities of coffee berries to normal roasts.

Caramelisation of the sugar, already added to the roasted nibs, imparts a characteristic flavour to the finished chocolate. This may be accomplished by heating the mixed chocolate paste in an iron "melangeur" or "conche" from 115° C. to 120° C., extending over a period of time of thirty to forty-five minutes, so that the whole is gradually and evenly cooked. Care must be taken that no chocolate adheres for any length of time to the hot, iron surface of the mixer, as a strong burnt flavour, of a most unpleasant nature, will result. When it is desired to secure this caramel flavour, the beans should only be semi-roasted, as the subsequent cooking develops the half-acquired aroma of the cacao at the same time as it caramelises the sugar. The operation of caramelisation may, with advantage, be conducted in the "conche" machine, already described in the chapter on "Milk Chocolate," and elsewhere, as the receptacles are especially adapted for heating to high temperatures. The rise of temperature should be gradual and evenly distributed over the vessel containing the chocolate to be cooked. The author, however, favours a long treatment in a "conche" machine at a comparatively low temperature, as the changes take place more gently and evenly, and there is less risk of spoiling the product. This question has been considered at some length in a previous chapter.

Similar, but, in our opinion, inferior, results are obtained

Fig. 20.—"Thermos" Caramelizing Machine. (See p. 346.)

By permission of Messrs. Sany, Janssens, et Cie., Paris.



by preparing, beforehand, the caramelised sugar, *i.e.*, sugar heated to  $182^{\circ}\text{C}$ ., and adding it to the cacao mass in the "mélangeur," or by carrying out the processes suggested in Chapter XIX.

"Thermos" (*Patent*) *Caramelising Machine* (Fig. 29). (*Messrs. Savy, Jeanjean & Cie., Paris.*)—The object of this machine is to transform the crystallisable sucrose in chocolate pastes into invert sugar by means of a high temperature, applied for a very short time. The purpose of the transformation is to prevent the power of absorption of liquid cacao butter by the sugar crystals which, by capillarity and the superficial area of the crystals, tend to dry up the mass by retention of the liquid fat on, and between, their surfaces. It is claimed, moreover, with every justification, that fine finish and perfect smoothness are also secured, and, though, undoubtedly, the flavour of the chocolate is also improved by the process, the makers of the machine depend more upon the blend of beans for the taste and aroma of the finished product.

The output of the machine is from 70—100 kilos *per* hour, and the product, obtained, possesses a velvety smoothness and is perfectly homogeneous.

The machine consists, principally, of a metal shell carrying on the inside a rotating drum of about 60 cms. diameter, which is ringed with channels about 3 mms. deep. A second drum, of slightly less diameter, is placed in front and below the first drum. A metal hopper feeds the chocolate paste between the two drums which, revolving at different speeds, carry the paste forward. The paste spreads over the whole surface of the larger drum as it is carried forward, and, owing to the thinness of the film, every portion is exposed to the hot air which is blown in, the surfaces both exposed to the air and to the drum being similarly heated. A temperature as high as  $120^{\circ}\text{C}$ . can be obtained.

A collecting knife with toothed blade is carried the whole length of the drum and scrapes off the heated paste which

falls into a channel, placed beneath, whence it is carried to the discharge of the machine by an endless screw.

There is no doubt that the machine is rapidly gaining in popularity, and, already, it can be found in many of the largest chocolate factories in Europe.

About 5 h.p. is required to drive the machine.

The roughness to the palate of the older-fashioned dry chocolate is due to the fact that the cacao mass and sugar are not refined to such an extent as in the smooth "fondant" chocolate, so popular at the present time. Close refining, whilst giving smoothness to chocolate, is apt to rob it of its aroma, especially if the finished article is allowed to lie about exposed to the air.

Chocolate, like flour and milk, readily takes up odours to which it is exposed, and it is, consequently, very necessary to prevent strong-smelling matters from coming into contact with the chocolate and from tainting the atmosphere of the rooms in which chocolate is stored.

Chocolate should never be packed direct in cardboard or plain paper, as the stale flavour, imparted, is most unpleasant, but all tablets should be wrapped in tinfoil previous to packing. In sampling chocolate, it will be found that tablets will have developed their aroma, for better or for worse, after two or three weeks; freshly moulded tablets never convey to the palate the true flavour of the chocolate.

#### **Flavouring Materials, such as Spices, Essences, etc.**

The principal flavouring materials, added to cocoas and chocolates, are vanilla or its substitute, vanillin. Cinnamon, nutmeg, cloves, cardamoms, coriander, balsam of Peru, gum benzoin, almonds, hazel-nuts, praliné, pine-nuts, coffee and milk are also commonly used.

Of these, by far the most popular flavour for plain eating-chocolate is vanilla.

*Vanilla and Vanillin.*—The vanilla of commerce is the seed pod of the parasitic orchid *Vanilla planifolia* which is cultivated in Mexico, Tahiti, Mauritius, Mayotte, Java, Seychelles and Ceylon.

The fresh pods do not contain much of the aromatic bodies characteristic of the vanilla beans of commerce, but these are developed in the subsequent treatment, of a fermentative character, which the beans are caused to undergo.

Fine vanilla beans come into the market, in tin boxes, as long moist black pods, usually covered with an efflorescence of white crystals. These crystals are aromatic and volatile, and consist of vanillin and a certain proportion of benzoic acid. During preparation, it is necessary to dry the beans, as not only do they rapidly develop mould, when moist, but, in the dry state, they better retain their aroma.

Inferior vanilla, from improper curing or from trees of other species of vanilla, bearing fruit of lower value, are frequently treated by rolling the pods in synthetic vanillin, benzoic acid, etc. For the detection of such fraud, the chemical reactions of the possible adulterants should be investigated.

From Brazil is obtained the species called "Vanillon," possessing a long bean of strong, but less aromatic, flavour.

Vanilla beans may be used either as "vanilla sugar," in which case the beans are cut up fine and mixed with sugar, and the whole reduced to a fine powder, or as extract, when an alcoholic solution is prepared by soaking the beans in the solvent.

Vanilla sugar.		Vanilla extract.	
Vanilla beans (fine ground)	1 part.	Vanilla beans (fine ground)	4 parts.
Sugar	5 parts.	Alcohol	5 "

The vanilla extract should be prepared by soaking the pods, finely crushed or ground, in alcohol for forty-eight hours, when the clear extract can be decanted or filtered off.

All vanilla preparations, as also all materials used for flavouring purposes, which owe their properties to volatile matters, should be kept in closely sealed vessels or bottles.

The objection to the use of "vanilla sugar," and of other preparations of the vanilla pod in which the whole bean is used, is the presence of the black specks or seeds which render unsightly the sugar or any light-coloured material, so flavoured. This objection does not hold good for cocoa and chocolate which are flavoured to the best advantage with vanilla bean. From the financial point of view, the use of synthetic vanillin, which, in recent years, has almost entirely replaced the vanilla bean for flavouring cocoa and chocolate, is to be recommended.

Vanilla beans seldom contain more than 2·5 per cent. of vanillin to which is principally due the aromatic property of vanilla. Vanillin was first synthesised in 1872 from oil of cloves, and the artificial product has steadily worked its way into the favour of the confectioner, both on account of its efficiency and low price *per* flavouring unit as compared with those of the natural bean.

The prices of vanilla beans and vanillin were, in 1911, 18s. and 12s. 6d. per lb., respectively. At the present time (early 1920), the prices are 15s. 6d. and 78s., respectively.

Vanillin sugar.		Vanillin extract.	
Vanillin	1 part.	Vanillin	1 part.
Alcohol (hot)	5 parts.	Alcohol (hot)	5 parts.
Sugar	20 „		

Vanillin is not so readily soluble in cold as in hot alcohol; consequently, in order to accelerate solution, hot alcohol, preferably, mixed with the vanillin crystals. In the preparation of "vanillin sugar," the dry, fine sugar is rubbed with the solution, added very slowly, and, when the whole is quite dry, it should be tinned up immediately, to prevent evaporation of the volatile aroma.



Vanillin has, approximately, forty times the flavouring power of vanilla; consequently, in the recipes, given, both the "vanillin sugar" and extract will be stronger than the corresponding preparation from the natural bean. The amounts of vanilla and vanillin, used in the preparation of "sugar" and extract, are, of course, optional and can be varied to taste and requirements.

The flavour of the bean is undoubtedly superior to that of its synthetic substitute, less harsh and more delicately aromatic, but the advantages to be obtained by the use of the latter are so great that they will outweigh the superior flavouring properties of the former, except in cases where the most delicate palates are to be pleased.

The handling of vanilla beans and vanillin is attended with some inconvenience, as the aromatic bodies they contain are most pungent and irritating to the nostrils. Moreover, they set up irritation of the skin and cause sores which, though not attended by danger, are extremely unpleasant and distressing.

*Almonds, Nuts, etc.*—Hazel-nuts and almonds are used extensively on the Continent for giving a slight nutty taste, though, when used in England, much larger quantities of nut-paste are added, and the compound article is sold under some fancy name, such as "Noisette," "Nut Chocolate," "Nuttis," etc. Whole roasted nuts are, frequently, embedded in the chocolate and enable a harder product, which still possesses the nutty characteristic, to be made. Particularly are "Almond bars," prepared in this manner, popular in America at the present time.

When nut- or almond-paste is added to chocolate mass, the melting-point of the mixture is considerably lowered from that of plain chocolate, owing to the presence of oil which the nuts contain. Consequently, a good nut chocolate is always **softer** than the ordinary plain variety and is more difficult **to handle** in the factory. Cacao butter substitutes, possessing

"hardening" properties, will, frequently, be found in the cheaper grades of nut chocolates. The same remarks apply to milk chocolates, though the presence of the large proportion of casein tends to give "body."

Soft chocolates of these varieties will not possess "snap," which is so characteristic of hard plain chocolate. "Snap" is due, usually, to the presence of a large proportion of cacao butter, which itself possesses a hard, crisp break, and to the thorough blending and working of the sugar and cacao mass in the "mélangeur" and other machines, during manufacture. This property may also be acquired by rapidly cooling the chocolate after moulding, and it is worthy of note that chocolate, cooled slowly, will never acquire "snap," however long it is allowed to remain. The addition of oil, the presence of air-bubbles in moulded chocolate, and insufficient working, will all tend to the reduction of "snap."

*Spices, etc.*—The spices—cinnamon, nutmeg, cloves, cardamoms, and coriander—may be added direct to the chocolate paste in the "mélangeur," though, owing to the gritty nature of ground spices and to the fact that a certain amount of sand is always to be found in them, their essential oils are employed, with advantage. Another point in favour of using the essential oils, in preference to the ground spices, is that a more constant and equal flavouring material can be prepared, as the natural spices fluctuate considerably in the potency of their aromatic contents.

It is not an uncommon occurrence to meet with ground spices, on the market, which have been partially deprived of their essential oils or mixed with starch, meal, etc., and spices, so treated, would, naturally, be of lower value for flavouring. The characters of the spices are too well-known to need description, and the quantity, to be added to chocolate, too much a matter of taste to justify a catalogue of recipes. It is not intended, therefore, to do more than outline the method of procedure for the preparation of spice flavourings.

If the powdered spices are used, it is advisable to wash, dry and grind the spices on the factory premises, as such a proceeding prevents any possibility of adulteration.

If the essential oils are employed, it is advisable, as with vanillin, to prepare an alcoholic solution, as, when the flavouring mixture is measured out, the errors which will occur in the volumes of many consecutive measurements will be of the less importance the greater the dilution. This error might be considerable, if the essential oil were used pure and undiluted; further, the advantages to be gained by making an alcoholic solution for the preparation of "cinnamon sugar" and "clove sugar," by the same method as "vanillin sugar," are such that less intimate mixing will be required, and less possibility of making serious error occurs. The addition of strong flavouring materials to a food preparation is one of those matters which, at present, must be left to the discretion of the human automaton, and, if errors are to be prevented, the processes must be rendered as "fool-proof" as possible, especially in a large factory. A 10 per cent. alcoholic solution of an essential oil is a convenient dilution for working, and, where used *per se*, should be added to the chocolate at as late a stage of its manufacture as possible, to ensure that the least amount of volatile flavouring matter escapes.

There are many chocolate improvers on the market, at the present, day but all those that have come under our notice have been prepared from essential oils of spices, extract of cacao shells or coffee, or blends of them.

*Coumarin and Heliotropin.*—Coumarin, or the flavouring material of the Tonquin bean, is very commonly used in cheap chocolate. Used, with nice discretion, in milk chocolate, it is not unpleasant, though, to the "connoisseur," the flavour is indicative of poor quality.

Heliotropin is used for the same purpose as coumarin, but it is less penetrating and rather more delicately aromatic.

*Gum Benzoin, etc.*—Balsam of Peru and gum benzoin, both being soluble in alcohol, are used in solution. The balsam derives its flavour chiefly from the esters of benzoic and cinnamic acids, but, owing to its strong and burning taste, must be judiciously added to chocolate which may be greatly improved by the presence of a small quantity.

Gum benzoin, in 25 per cent. alcoholic solution, is principally used for chocolate varnish, the highly shining surface of decorative chocolate work or exhibition chocolate, that is required to maintain its lustre while exposed, being obtained by a solution of this gum.

*Coffee.*—The addition of roasted coffee to chocolate gives a very fine flavour, and, if used in extremely small quantities, is difficult to detect as the flavouring material.

The coffee should be added to the chocolate mass running in the "mélangeur" for the first time, as the hard and gritty nature of the ground and roasted coffee makes it desirable that it should be refined at least as often as the chocolate. Three parts of ground and roasted coffee to 100 parts of chocolate mass is a good proportion, though, to increase the flavour, as much as 5 per cent. of coffee may be used. Higher than 5 per cent., the colour of the chocolate will suffer, and the grittiness, due to the coffee, may be felt.

The trouble of grittiness may be overcome by steeping the coffee-grains, contained in a muslin bag, in melted cacao butter, which extracts all the aromatic oil. This, however, cannot be recommended as an economical method, seeing that little or no "return" in weight is obtained. Coffee extracts may also be used and, if prepared fresh from freshly roasted and ground coffee, will give good results.

It is worthy of note that, with the high price of alcohol at present obtaining, many of the essential oils will be found to be soluble in vegetable oils and fats such as arachis oil and cacao butter, which may be employed, instead of alcohol, with considerable benefit to the manufacture.

## CHAPTER XXIV

### NUTRITIVE VALUE AND DIGESTIBILITY OF COCOA PREPARATIONS

It has seemed desirable to include a chapter on the nutritive value and digestibility of cocoa and its preparations, seeing that much misleading information has been published in medical journals.

Already, some points have been considered in the *Introduction*, and it is not intended to cover this ground again. There is, however, a certain truth underlying the objections raised to cocoa by physicians from time to time, though, as in all physiological tests, it is not easy to prove, by facts and figures, statements that have been quoted from very earliest times. That the benefits outweigh the objectionable features of the preparations there can, however, be no shadow of doubt.

Thomas Gage (1648), in his work, quoted later more fully, states: "This cocoa though, as every simple, it contains the qualities of the foure Elements, yet, in the opinion of most Physitians, it is held to be cold and dry. It is also in the substance that rules these two qualities, restraining and obstructive, of the nature of the Element of the Earth."

Gerard, in his "Herbal" of 1636, just mentions cacao, but only as a drink which they "highly esteem in America" and which is of an "astringent and ungrateful taste." Culpepper is silent on the subject.

With the exception of the attacks made by Franciscus Rauch in 1624, the earliest reference, yet found, to the consideration of the physiological aspect of cocoa prepara-

tions, Gage's work appears to be the first attempt to study the subject scientifically. Indeed, we learn from the "Natural History of Chocolate, a pamphlet published by Christopher Wilkinson at the Black Boy over against St. Dunstan's Church in Fleet-street," 1682, that Mr. Gage "drank chocolate in the Indies two or three times every day for twelve years and he scarce knew what any disease was all that time, he growing very fat." Wilkinson, himself, had eaten great quantities of the kernels raw "without the least inconvenience," and writes that he had heard "that Mr. Boyle and Dr. Shibbs have let down into their stomachs some pounds of them raw without any molestation; the stomachs seemed rather to be satisfied than cloy'd with them which is an argument they are soon dissolved." We know, also, that the Spaniards did not scruple to eat cocoa upon their great fast days.

But, in early times, when chocolate was looked upon by the American native *habitués* as a stimulant food and by the wealthy European as a luxury with certain "secret" powers, it is not surprising to learn that, like all stimulants, it was recommended by the Herbalists and Physicians for application according to the constitution of the consumer. Thus—"In cold constitutions, Jamaica Pepper, Cinnamon, Nutmegs, Cloves, etc., may be mixt with the cacao Nut . . . In hot Consumptive tempers you may mix Almonds, Pistachios, etc. . . . and sometimes Steel and Rhubarb may be added for young green ladies." Wilkinson makes a naïve suggestion that, had Rachel known of the existence of chocolate, she would not have purchased Mandrakea for Jacob.

W. Hughes, in a book entitled "The American Physitian or a Treatise of the Roots, Plants, Trees, Shrubs, Fruit, Herbs, etc., Growing in the English Plantations in America, 1672," has provided a lot of information on the virtues of chocolate and has stated: "Chocolate is most excellent in nourishing and preserving health entire, purging by Expec-

torations, and especially by the sweat vents of the body, preventing unnatural fumes ascending to the head." His references to the properties of the "oil" will be given later.

Yet another word of praise for chocolate is given by one, Dr. Doran, who says, in his "Table Traits with something on Them,"—"I must not omit to mention that the favourite beverage of Voltaire at the Café Procope (Paris) was *choca*: a mixture of coffee (with milk) and chocolate. The Emperor Napoleon was as fond of the same mixture as he was of Chambertin and, in truth, I do not know a draught which so perfectly soothes and revives as that of hot well frothed *choca*."

From Robinson's work, "The Early History of Coffee Houses in England," (1893), are taken the following lines, which suggest that the beverage was less popular with the lower classes, at any rate in this country, at that time:—

" The Player calls for Chocolate.  
At which the Bumpkin, wondering at,  
'Cries, ' Ho, my Mist'ers, what d'ye speak,  
D'ye call for a drink in Heathen Greek ?  
Give me good old Ale or Beer,  
Or else I will not drink I swear."

We were ever a conservative race !

We have occasion to refer, later, to "The Indian Nectar or a discourse concerning Chokolata," by Henry Stubbe and Thomas Lord Windsor, dated 1662. These two enthusiasts had collected recipes for the making of chocolate and had placed them in the hands of "an honest though poor man, Richard Mortimer, of Sun Alley in East Smithfield," but, evidently, they were a little doubtful of the effects that their preparations would have upon the public, for they write: "He will attend on my Physician of note to receive his directions (in experiments) and also to inform him if he would vary from my ways by any addition, etc." And again, later, they write: "and, as I shall endeavour wholly to oblige

mankind, with further Observations and Enquiries concerning Chocolata so I hope my example will stir up others to the like performances, and that persons of ingenuity will either publish their observations or direct them to me and leave them either with the Lady Windsor or with Mr. Andrew Crook . . . to be sent to me that I may not be ignorant of what effects Chocolata or its particular Ingredients have here in England."

It seems that the idea that cocoa preparations affected the sexual organs was of early origin, for, besides the work of Ranch, 1624, already mentioned, and the article in the *Spectator*, 1712, Stubbe discusses in his book the physiological functions of his chocolate and propounds a hypothesis which will dispense with sexual function. The modern physician forbids chocolate and cocoa, except in very small quantities, in cases of bladder affections, such as cystitis, owing to the irritability of its infusion upon that organ, a fact which, probably, gave rise to the earlier suggestions.

To descend to more recent times, an excellent article, if somewhat unscientific from the chemical standpoint, was published in the *Lancet*, January 7th, 1905, entitled "Cocoa chemically and physiologically considered." The clue to the purpose of the article is found in an early paragraph: "The present popularity of cocoa as a beverage and the fact that so many give it preference to tea and coffee raise so important physiological questions which claim more attention than they have hitherto deserved." After considering the merits and the demerits of the three beverages, the amounts of tannin and alkaloid they contain and their presumed digestibility, some actual digestion experiments, performed *in vitro*, are described. It is with these latter that we are principally concerned here.

It is unfortunate that such a statement as: "the development of the flavour and the peculiar character of coffee is brought about by merely roasting, but in tea and cocoa both



a fermentation process and a species of roasting are employed to the same end " should have been allowed to stand without further comment, since coffee also is fermented, and we know that prolonged fermentation is detrimental to the aroma. Nevertheless, the article is full of sound information, even if it has to be selected for reproduction here, of a nature that we have not been able to find elsewhere.

Analyses of cocoas from the leading firms are given as :—

*Analyses of Cocoa Powders.*

	Per cent.
Moisture . . . . .	3.0 to 8.0
Nitrogenous matter ( $N \times 6.3$ ) . . . . .	19.0 to 20.0
Fat . . . . .	26.0 to 31.0
Mineral matter (ash) . . . . .	3.9 to 8.8
Starch . . . . .	2.7 to 7.3
Cellulose . . . . .	6.8 to 7.2
Other non-nitrogenous matters . . . . .	29.0 to 31.0
Theobromine . . . . .	1.7 to 2.0

The degree of fineness to which the cocoa is ground is a matter of considerable dietetic importance, as will be seen later, chiefly because, when placed in water, an almost complete suspension is secured, and no sediment settles out, whilst the digestive juices are enabled to have full play on each minute particle taken into the stomach.

It was shown that cocoas were usually acid in reaction and that, when acid, the tendency to settle out in the cup was most marked. By the addition of "natural salts," the cocoa became "not acid" to test papers, "owing to the action of alkaline organic salts, and yields practically no sediment in the cup, the exceedingly fine particles being in a state of perfect suspension, a condition which is assisted by the salts." This, of course, is known in the trade as rendering cocoa "soluble," and is actually brought about, in this country and all over Europe and America, by the addition of

alkalis. With this, however, the writers of the article strongly disagree, but, without wishing to be disrespectful we can only impute their denial to ignorance of the trade and to chemical methods. Passing over these verbal differences of opinion, we come to another table which is distinctly interesting as showing that, when addition of alkali is made alkalinity appears only in the ash and not in the cocoa powder, as such. In spite of this fact, we must again differ from these writers who say : “ It has been freely stated that some cocoas in the English market are largely manipulated with alkali. Our experience gives no support whatever to such a statement,” since they have fallen into an elementary error of attributing the alkalinity of the ash to the *increase* of “ natural organic salts of the cocoa ” and not to the *addition* of alkalis which, possibly, may have neutralised the organic acids of the cocoa. Yet, they are willing to grant that the iniquitous habit of adding alkali to cocoa is practised in Germany. It is, indeed, often difficult to discriminate between scientific facts and medical fancies in this paper.

For the experiments on digestibility of cocoa in artificial gastric and pancreatic juices, Cadbury's cocoa was taken as an example of cocoa prepared from cacao beans simply by roasting, grinding and pressing, to remove a certain quantity of fat ; Van Houten's cocoa was selected as an example in which “ natural salts are increased,” and Plasmon cocoa as representing a powder containing added matter in the form of dried milk solids (approximately sixty parts milk solids to forty parts cocoa powder), which should be soluble. The following figures were obtained :—

Cocoa.	Total dissolved matters.	Mineral matters.	Organic matters.	Proteid
Cadbury's	19·80	3·20	16·60	5·90
Van Houten's	23·00	5·20	17·80	10·55
Plasmon	50·40	5·00	45·40	38·21

The increased solubility, by the use of alkali, of Van Houten's cocoa over that of Cadbury will be noticed in Column I. Further, Van Houten's cocoa was observed to be exceedingly finely ground, "in fact comminuted." So far as the proteids are concerned, Cadbury's cocoa yielded up to hot water 33 per cent. of its proteid matter, Van Houten's cocoa 60 per cent. and Plasmon cocoa 70 per cent., the total proteids being 18.60 per cent., 17.25 per cent., and 54.43 per cent., respectively.

The amount of sediment, settling out, was next measured, one gramme of each cocoa being boiled with water and made up to 100 ccs.

Cadbury's cocoa	.	5.0 ccs.	sediment after half-hour.
Van Houten's cocoa	.	1.0 cc.	" " "
Plasmon cocoa	.	3.0 ccs.	" " "

Evidently, and as might have been expected, Plasmon, which was regarded as a soluble form of milk proteid, is rendered to some extent insoluble when added to cocoa. As a matter of fact, the present writer, speaking from experience with this product, can assert that Plasmon was seldom totally soluble in water and that the addition of alkali did, of course, greatly increase its solubility, whilst the presence of tannin in the cocoa will always render a certain amount of milk solids, when present, insoluble in water.

The experiments with the same cocoa powders and artificial gastric and pancreatic juices are very interesting. The cocoas were prepared (as directed by the manufacturers) by treating with boiling water. The infusion was brought to 100° F. (approximately the normal temperature of the human body); and a definite volume of digestive fluid was added. In the cases of gastric digestion, the mixture was acidified with hydrochloric acid, and the pancreatic fluid was rendered alkaline with sodium carbonate. Digestion was carried out

for four hours at a constant temperature, and the mixture was occasionally stirred. The following figures show the percentage amount of the total proteid hydrolysed by the ferments, respectively, after four hours :—

Cocoa.	Gastric digestion.	Pancreatic digestion.	Undigested proteid.
Cadbury's	40·45	31·40	28·15
Van Houten's	67·20	4·11	28·69
Plasmon	37·50	56·20	6·30

These figures, taken in conjunction with those already given, speak for themselves.

It is pointed out by the authors that, in the literature on the subject, the amount of proteid, found by other observers in cocoas, is generally in excess of the truth, for the reason, probably, that the amount of proteid has been based on the total nitrogen found, whilst the theobromine (amounting to about 0·5 N per cent.) should have been deducted before the calculation for proteid was made. We do not think, however, that this is likely to have occurred in many chemical laboratories, of any repute. It can be readily understood why the milk proteids of Plasmon cocoa are found to be digested in alkaline solution ; indeed, we should have been surprised had it not been the case, though the fact seems to have caused the original writers some surprise.

As the undigested matter contained practically all the fat, since the action of artificial pancreatic juices upon fat is very feeble, the figures were recalculated as fat-free residue. Thus :—

Cocoa.	Undigested.	Digested by difference.
Cadbury's	31·73	68·27
Van Houten's	31·65	68·35
Plasmon	14·88	85·12

The last experiments, made, were upon the same cocoas, but with weak acids and alkalis in the absence of ferments.

Cocoa.	Soluble in weak acids.	Soluble in weak alkalis.	Insoluble in both.	Total solubility.
Cadbury's	41.50	51.70	6.80	93.20
Van Houten's	41.10	51.70	7.20	92.80
Plasmon	58.10	39.60	2.30	97.70

In the figures shown in the first column, 7.33 per cent. of starch for Cadbury's cocoa, 2.70 per cent. for Van Houten's cocoa and 1.99 per cent. for Plasmon cocoa, must be included, whilst, in the insoluble figures, the fibre will be found.

Many references to chocolates have appeared in the *Lancet*, from time to time, but, as a rule, they are more advertisements of the product than attempts to provide physiological data.

In 1910, Albahary (*"Ann. Falsif,"* iii., 159—163), pronounced judgment on alkali-treated "soluble" cocoas. He found that the so-called "soluble" cocoas were not more soluble than natural cocoas from which the greater portion of the fat had been expressed. The larger quantities of alkali, soap and colouring matter, going into solution during infusion, caused the fat of the treated cocoas to be more perfectly emulsified and the insoluble constituents to remain for a longer time in suspension. The same writer is emphatic in his denial that "soluble" cocoas are more easily assimilated than the natural cocoas and that the proteids and other substances, present in the former, are more soluble than those in the latter. Further, the presence of soap, resulting from the neutralisation of the fatty acids of the cacao butter, is particularly objectionable when the cocoa is consumed by children and invalids. The writers in the *Lancet* in 1905 have stated, however, that no soap could be detected in so-called "alkalised" cocoas.

It has been estimated that cacao, which yields 579,000

calories, falls to 339,000 calories heat value, if it is expressed so as to contain 15 per cent. of fat. By defatting the cacao, non-digestible albuminoids rise from 8.2 per cent. to 14.9 per cent., and cacao fibre from 3.4 per cent. to 6.2 per cent. On the other hand, the process of expression of fat raises the theobromine value appreciably.

It has already been pointed out that the alkalis, when used correctly, are not present in cocoa powder as free alkali, but in combination with phosphoric and organic acids; in other words, the salts of the alkali are in a form usually consumed by man in his ordinary food. Man consumes, daily, some 4—5 grammes of alkali compounds (calculated as  $K_2O$ ) in his food, and the proportion added, if a cup of alkalisied cocoa is also taken, amounting to a further 0.07—0.10 grammes of  $K_2O$ , is insignificant.

Forster \* estimated that 90 per cent. of the dry matter of cocoa was readily digested, 80 per cent. of the nitrogenous matters and 100 per cent. of the fatty and mineral matters being easily assimilated.

Neumann † has made a very complete study of the digestibility of cocoa powders by studying the effect of a diet of cocoa, with and without other foodstuffs, on the weight of the human body, and by analyses of the *excreta*. Albuminoids are absorbed, apparently, to the extent of a minimum of 45 per cent., when cocoa, alone, is used as diet. The addition of cocoa to other articles of food seems to reduce the total amount of nitrogen absorbed, and the nitrogen absorbed is also dependent upon the amount of cacao fat present, the less the fat the less are the nitrogenous bodies assimilated.

Cacao butter was shown to be absorbed to about 95 per cent., when consumed with other foods, the normal fat of

\* J. Forster, *Hyg. Rund.*, 1900, 305.

† Neumann, "Die Bewertung des Kakaos als Nahr und Genussm.," R. Oldenburg, Munich, 1906.

normal foods being absorbed to about the same amount. But, when consumed alone in cocoa powder, cacao butter showed an assimilation of only 87.1 per cent., as against 89.6 per cent. when mixed, as cocoa powder, with other foods. It was shown that a cocoa powder containing about 30 per cent. of fat was the most easily assimilated.

Gautier \* has stated that 100 grammes of cocoa powder, defatted to 25 per cent. of fat, contains 1.3 grammes theobromine, 17 grammes of nitrogenous matters of which 8 per cent. is albumen, and 10 to 12 grammes of carbohydrates. In one cup of cocoa containing 10 grammes of powder, one-tenth of the quantities, shown, will be consumed. Gautier concludes that the stimulating effect on a tired person of a cup of chocolate containing 14 grammes of sustaining matter, of which 7 grammes are sugar and 2.5 grammes are fat, can only be explained by an effect on the nervous system, provoked by the aroma of the cocoa and the tonic influence of the theobromine, whilst the nutritive value of the preparation has, in addition also, an accumulative effect, in proportion as it is absorbed.

With regard to cacao butter, Hughes, in the very early treatise already mentioned, says: "Cacao Oil is internally administered, it is good against all Coughs, shortness of breath, opening and making the roughness of the arteries smooth, palliating all sharp Rheums and contributing very much to the Radicle Moisture, being very nourishing and excellent against Consumption," and, again, that it can be "effectually externally applied against all Inflammations, *i.e.*, Phlegmons, Erisipelas, S. Anthony's Fire, Scaldings and Burnings, the place being annointed therewith." Were it not for the quaint wording, these lines might have been taken from the most recent United States Dispensatory, which recommends the use of cacao butter for a number of similar complaints.

As to the digestibility of cacao butter, however, there is

\* Armand Gautier, "L'Alimentation, etc.," 327 *et seq.*

more to be said, the work of Gardner and Fox \* being the most recent attempt to reach a conclusion. A good *résumé* of the previous work on the subject is given by these workers and is worthy of study by those requiring further information.

It has already been stated that cacao butter is resistant to enzyme action *in vitro*, but that it is easily digested when the cocoa, itself, is eaten. In 1895, Cohn †, in experiments carried out on himself, found that 95 per cent. of the fat of cacao was absorbed, but only half the proteids. In 1906, Neumann ‡ obtained an absorption figure of 90 per cent. for the fat in prolonged and favourable experiments, and similar results were obtained by Gerlach, § in 1907. In the same year, Pincussohn, || experimenting on a number of subjects, found an absorption figure for cacao butter as high as 98 per cent., whilst the proteid absorption varied, considerably, between 60 per cent. and 90 per cent., but was best in those preparations containing a low percentage of fat.

None of these observers noticed any ill effects upon the health as a result of taking quite large quantities of cacao butter over considerable periods of time. Zuntz, ¶ working with cacao butter in eleven experiments, only obtained a figure of 89·6 per cent. utilisation, the actual figures varying from 79·1 per cent. to 95·5 per cent. This large variation was probably due to the fact that, to some of the subjects, cacao butter was actually nauseating, causing "loss of appetite," "headache," "loss of ambition" and sleeplessness. Finally, as a result of eating 109 grammes *per day*, undesirable physiological derangements were experienced. The limit of tolerance, according to these experiments, lay between 51 and 109 grammes *per day*.

\* J. A. Gardner and F. W. Fox, *Biochem. Jour.*, 1919, xii., 368—377.

† H. Cohn, *Zeitsch. physiol. Chem.*, 1895, xxi.

‡ Neumann, *Arch. Hygiene*, lviii., 1.<sup>o</sup>

§ V. Gerlach, *Zeitsch. physik diät Therapic*, 1907, xi., 204.

|| L. Pincussohn, *Zeitsch. klin. Med.*, 1907, lxiii., 450.

¶ N. Zuntz, *Therap. Monatsch.*, 1890, iv., 471.



The general conclusions, reached by Gardner and Fox, were somewhat of the same order, though more than one of the subjects took quantities of cacao fat up to 237 grammes in a five-day test, without any unpleasant symptoms. The same writers concluded that cacao butter was rather less digestible than cow's butter, but that the experiments showed satisfactory utilisation, except when excessive quantities of cacao butter, which had a slight laxative effect, were consumed. There was no indication that cacao butter was a "slow poison."

In the *British Food Journal*, April, 1916, an interesting article appears on "Cocoa and Chocolate." Many inaccuracies have been allowed to stand, such as: "There is, however, no nitrogen except in the form of theobromine, which is a mild stimulant and has also a steadying action on the digestion but does not supply nitrogen for building up tissue," and, again: "the only possible use for husks seems to be to burn them and so economise coal." On the other hand, the article contains a story relating to the evil effects produced by the inclusion of husk in cocoa preparations, which, presumably, has some foundation in fact.—"An interesting case of the adulteration of cocoa with husks, or, to be more accurate, of adulterating ground husks with cocoa, has recently come to light in Germany and incidentally affords an illustration of German morality in trade matters. A large number of severe cases of dysentery and gastritis occurred among the German troops at certain points on the front, and the medical officers there and at the base were at the first quite at a loss to explain them. They were subsequently found to be confined to soldiers who had eaten certain chocolates sent to them as love-gifts (*liebesgaben*)\* by their civilian friends. These love-gifts were traced by the police to a Hamburg firm which made a speciality of making

\* This word is correctly translated as "comforts for the troops," and not "love-gifts."

and advertising war chocolates for presentation to the men in the trenches. Quantities of these chocolates were bought by the authorities, and the cocoa part of them was found to consist almost entirely of husk. The next step was naturally to raid the firm's premises, where huge quantities of these chocolates, ready for sale, were seized, together with no less than 50 tons of crude husk purchased in Germany and also in Holland. We have not yet heard what happened to the firm."



## **PART III**

### **CHEMISTRY OF CACAO**

#### **A. SURVEY OF THE COMPONENTS OF CACAO, COCOA AND CHOCOLATE**

#### **B. METHODS OF ANALYSIS**



## CHAPTER XXV

### A.

#### SURVEY OF THE COMPONENT PARTS OF ROASTED CACAO BEANS—HUSK

ROASTED cacao is divided up during the process of manufacture of cocoa and chocolate into (1) the husks or shells, which are the by-product, of little value; (2) the nibs, which, as has been seen in the foregoing chapters, should comprise the base for sweet and fancy chocolates, and which, when partially deprived of fat for cocoa powders, constitute the most commercially important fraction of cacao.

#### Husk.

(8 per cent. to 20 per cent. of the whole bean.)

König has shown the amount of husk, determined by Laube and Aldendorff, to vary considerably with the origin of the bean. This is not remarkable when the different processes which the beans undergo in fermenting and curing in various localities, previously described in Chapters VI. and VII., are considered. The following table (Table LVI.) shows two sets of results obtained by different authors on various cacaos.

The considerable variations which occur between the figures of different observers are attributable to the varying proportions of sand and earth adhering to the beans. Such differences frequently occur between the figures of the same variety of bean sold under the same mark.

TABLE LVI.—*Percentage of Husk in Cacao Beans.\**

	I.	II.	III.
Caracas . . . . .	20.09	15.00	14.20—20.10
Guayaquil . . . . .	—	18.68	11.20—14.50
Trinidad . . . . .	14.04	14.68	11.23—15.65
Puerto Cabello . . . . .	14.92	12.28	13.50
Soconusco . . . . .	18.58	—	—
Surinam . . . . .	—	14.60	—
Machala . . . . .	—	16.14	—
Port au Prince . . . . .	—	16.00	—
Accra . . . . .	—	—	9.77—12.24

I. König, *Die mensch. Nahr. u. Genussm.*, i., 261.

II. Zipperer, "Untersuchungen über Cacao, etc.," 55.

III. Whympre and Bradley. Unpublished, 1920.

The chemical composition of husks from cacao of different varieties can be seen in Table LVII.

TABLE LVII.—*Composition of Husk.†*

	I	II	III.	IV.	V	VI	VII	VIII
Water . . . . .	12.57	5.12	—	—	9.30	12.51	4.50	7.83
Fat . . . . .	3.30	12.92	3.6	—	3.83	4.23	4.40	6.38
Ash . . . . .	7.35	6.92	5.7	6.46	8.26	10.20	7.30	7.12
Nitrogen . . . . .	—	2.63	2.05	—	3.00	2.19	2.50	—
Proteid . . . . .	14.69	16.44	12.8	—	18.81	13.69	—	14.29
Fibre . . . . .	16.33	13.17	—	13.05	13.85	16.71	14.00	14.69
Theobromine, caffeine.	0.79	—	0.58	—	—	—	—	—

I. G. Paris, *Zeitsch. Nahr. Genussm.*, 1898, vi., 389.

II. F. T. Schutt, "Annual Rep. Exptl. Farms (Canada)," 1898, 151; 1899, 851.

III. S. Dekker, *Chem. Centr.*, 1902, ii., 1217.IV. H. Luhrig, *Zeitsch. Nahr. Genussm.*, 1905, ix., 263.V. A. Smetham, *Jour. Royal Lancs. Agric. Soc.*, 1909.

VI. Zipperer, "Untersuchungen über Kakao, etc.," 55.

VII. N. P. Booth, *The Analyst*, 1909, xxxiv., 144.VIII. König, *Die mensch. Nahr. Genussm.*, i., 261.

Besides these analyses, there are many other contributions to our existing knowledge of the component parts

\* *Vide* also Chapters VIII. and IX.† *Vide* also Chapter IX., Tables XVIII. and XIX.

of cacao husk. The researches of Matthes \* and his co-workers are worthy of comment, as also are those of Filsinger † and Welmans, ‡ and, more recently, Baker and Hulton. §

In the foregoing table, Luhrig's figures are calculated on the dry substance. Paris', Schutt's and Smetham's results were obtained in ascertaining the food value of cacao husk, a waste product of the chocolate factory, for cattle food. The utilisation of the husk for the preparation of a marketable product would be of great value to the cocoa and chocolate manufacturer, and the subject has occupied the attention of many chemists, agriculturists, and others.

Knapp || has considered this question, as well as Baker and Hulton, during the years of war, when both the regulations of the Food Controller and the appearance on the English market of "cocoa teas" necessitated investigation into the food-value of cacao husks. In 1910, a cocoa powder containing 18 per cent. of shell was found to be genuine by law. In 1918, the "Cocoa Powder Order" regulated the manufacture and sale of cocoa powders, substantially as follows :—

- (1) "Grade A cocoa powder" was to contain not more than 2 per cent. of cacao-bean shell, and could be sold as such, provided that not more than 60 per cent. of the cocoa powder, sold by the manufacturer in any month, was of this quality, and, also, provided that the powder, sold, was packed in cartons, or was consigned to a person authorised by the Food Controller to purchase "Grade A cocoa powder" not packed in cartons. The maximum price, fixed for

\* Matthes, etc., *Ber.*, 1907, xv., 4195, and *Zeitsch. Nahr. Genussm.*, 1906, xii., 159, etc.

† Filsinger, *Zeitsch. öffent. Chem.*, 1899, etc.

‡ P. Welmans, *ibid.*, 1901, vii., 491.

§ J. L. Baker and H. F. E. Hulton, *The Analyst*, 1918, xliii., 189—197, 507.

|| A. W. Knapp, *Jour. Soc. Chem. Ind.*, July, 1918 (T.), xxxvii., 240—242



"Grade A," was 3*s.* 2*d.* *per* pound, and the packets were to be suitably labelled to show the grade of cocoa, enclosed.

(2) "Grade B cocoa powders" were defined as cocoas containing not more than 5 per cent. of cacao-bean shell (and no cocoa powder should be manufactured that contained more than this amount); and not less than 22½ per cent., and not more than 30 per cent., of cacao butter. This cocoa had to be invoiced as "Grade B cocoa powder," and was to be packed in barrels or cases, unless, at the buyer's request, the manufacturer packed the cocoa powder in cartons, which had to bear the words "Grade B cocoa powder." The maximum price, fixed for "Grade B cocoa powder," was 2*s.* 2*d.* *per* pound, or, if sold in cartons, 2*s.* 6*d.* *per* pound.

(3) Part 3 of the Order provided that no cacao shell, or any mixture (other than chocolate) which contained more than 5 per cent. of cacao shell, could be sold by retail, on and after April 8th, at a price exceeding 6*d.* *per* pound.

Knapp calculated from the official returns on cacao that the world production of cacao shell was about 36,000 tons *per annum*, of which Europe was responsible for 22,000 tons. Great Britain, in 1916, provided 4,773 tons.

The average amount of shell in cacao beans is about 12·5 per cent. According to Knapp, and confirmed by the author's experience, some 10½ per cent. of shell is easily removed. Of the "smalls," which approximate 4 per cent., about 36 per cent. is shell, and, owing to the losses on roasting, separating, etc., only about 78·5 per cent. of usable nibs are recovered from the original cacao, and of this about 2 per cent. is shell, which is only with the greatest difficulty removed. The price for cacao shells rose rapidly during the war; thus, in 1912, the price *per* ton was 65*s.*; in 1913, 1914 and 1915, 70*s.*; in 1916, 90*s.*; in 1917, 128*s.*; whilst in May, 1918, 310*s.* was quoted.

The variations in price, at any one time, for cacao shells

depend upon the amount of nibs left behind in the so-called "offal." Knapp quotes for shell, in 1916, containing less

TABLE LVIII.—Analyses of "Cocoa Teas."  
(Baker and Hulton.)

	Cocoa teas.				Cacao shell.	Cacao nib.	Cocoa (alkalised).
	A.	B.	C.	D.	Average.	Average.	Average.
Moisture . . . . .	11.0	10.8	8.76	10.44	4.68	3.0	5.68
Fat . . . . .	4.0	20.05	12.65	4.7	3.56	50	24.0
Total ash . . . . .	15.0 <b>17.64</b>	8.0 <b>11.6</b>	7.48 <b>9.52</b>	10.04 <b>11.8</b>	10.52 <b>11.46</b>	<b>3.21</b> <b>6.83</b>	6.78 <b>9.67</b>
Ash soluble in water . . . . .	2.2 <b>2.59</b>	5.78 <b>8.38</b>	5.14 <b>6.52</b>	6.68 <b>7.86</b>	4.2 <b>4.58</b>	1.21 <b>2.57</b>	5.2 <b>7.43</b>
Ash insoluble in hydrochloric acid . . . . .	5.14 <b>6.05</b>	.36 <b>.52</b>	.34 <b>.43</b>	.5 <b>.59</b>	2.5 <b>2.72</b>	.04 <b>.08</b>	.2 <b>.29</b>
Soluble ash per cent. on total ash . . . . .	14.8	72	69	66.5	45.5	38	76
Alkalinity of water-soluble ash as K <sub>2</sub> O . . . . .	.33 <b>.39</b>	3.85 <b>5.58</b>	3.08 <b>3.92</b>	4.28 <b>5.04</b>	2.52 <b>2.75</b>	.85 <b>1.81</b>	3.03 <b>4.34</b>
Water-soluble K <sub>2</sub> O per cent. on total ash . . . . .	2.2	48	41.2	42.6	28.1	26	45.7
Nitrogen . . . . .	2.03 <b>2.39</b>	2.38 <b>3.45</b>	2.38 <b>3.03</b>	2.66 <b>3.13</b>	2.42 <b>2.64</b>	2.3 <b>4.9</b>	3.06 <b>4.87</b>
Crude protein (N 6.25) . . . . .	12.69 <b>14.9</b>	14.87 <b>21.55</b>	14.87 <b>18.9</b>	16.62 <b>19.55</b>	15.1 <b>16.52</b>	14.37 <b>30.6</b>	19.1 <b>27.3</b>
Crude fibre . . . . .	12.8 <b>15.0</b>	10.8 <b>15.65</b>	13.8 <b>17.5</b>	16.6 <b>19.5</b>	15.42 <b>16.8</b>	2.68 <b>5.7</b>	4.3 <b>6.14</b>
Matter soluble in cold water . . . . .	21.7 <b>25.5</b>	17.5 <b>25.36</b>	20.2 <b>25.6</b>	22.0 <b>25.9</b>	21.0 <b>23.0</b>	11.4 <b>24.2</b>	19.5 <b>28.0</b>
Matter soluble in hot water . . . . .	27.3	28	34	31.8	—	—	—
Levigation sediment . . . . .	—	—	—	—	27.6 <b>30.0</b>	1.41 <b>3.0</b>	—
Price per pound (March, 1918) . . . . .	6d.	1s.	1s.	1s. 4d.	2d. or 3d.	—	2s. 8d.

"NOTE.—The figures in heavier type are percentages on dry fat-free matter. The values given for average cacao nib are chiefly based on Winton, Silverman, and Bailey, and Booth, Cribb, and Richards (*loc. cit.*). The alkalised cocoa in the last column was a sample analysed by ourselves."

than 1 per cent. of nibs, 120s. ; shell with 2·8 per cent. nibs present, 130s. ; shell with 40 per cent. nibs present, 150s. ; and shell with 15 per cent. of nibs present, 200s.

The use of "cocoa teas" in Ireland is well established, but it was only during the War that such preparations as "Celesco," and other cacao shell products, found their way into the English markets. Baker and Hulton have provided analyses of some "cocoa teas" of sufficient interest to be included in this place (though we are inclined to differ from some of their observations, especially on sample B.), which are given above.

"A. An inspection of the analytical values for this material indicates clearly that it consists of shell without any admixture of nib, the outstanding point of interest being the remarkably high ash content, a large proportion of which is insoluble in hydrochloric acid. The sample of which it is composed was 'earthed,' and it is probable that a considerable amount of finely-divided mineral matter would be present in the emulsion prepared from it, and would, therefore, be consumed by any one drinking the 'tea' prepared from it.

"B. This sample, as we have just shown in the worked example given above" (the calculation is considered later in the present work), "consists of a mixture of about 64 per cent. of shell and the balance (36 per cent.) of nib. The analytical values also show that the beans of which it is composed have been roasted with a soluble alkali; it contains the highest proportion of nib of any of the four samples analysed.

"C. This sample is also a mixture of shell and nib, the percentage of the former being about 86 per cent., the balance (14 per cent.) being nib. Like B., it has also been treated with alkali.

"D. The values found for fat, crude fibre, and water-soluble matter, leave little room for doubt that

no nib enters into the composition of this high-priced material."

For a more balanced view of the supposed alkali treatment, the work of Arpin,\* to which already full reference has been made in a previous chapter, should be consulted.

Dubois has sent to Knapp the following figures, obtained in America, showing the digestible nutrients in 100 lbs. of shell:—Proteid, 1.53; fibre, 6.45; N.-free extract, 40.6; fat, 4.91; fuel value, 111,079 calories, or 1 lb. of shell to 4,404 B.Th.U.

Knapp also mentions that successful feeding experiments with cacao shell have been conducted in Turin, Germany and America, and quotes a case of poisoning of cattle in Germany in 1916, supposed to have been due to cacao shells. In view of the numberless successful feeding trials that have taken place, it is more probable that too high a percentage of cacao shell was included, rather than that the poisoning was due to theobromine, as was suggested at the time.

The introduction of cheap oil-cakes had somewhat limited the possibilities of using cacao husk for cattle food, and it was only during the War that experiments were again made on a large scale. Some excellent results have been obtained with cacao-husk cake on experimental farms. The feeding of cattle, milch cows, etc., on cacao husk, made up in a similar way to the modern oil-cakes, has shown that the animals thrive on the diet and put on weight. The amount of cream yielded by the milk of cows, fed on cacao cake, was found to be larger than from grass-fed animals, and, after a prolonged treatment, the quantity of milk *per* cow was noted to increase sensibly also.

The results of experiments can be found in the records of the Society of German Chocolate Makers,† the annual

\* Arpin, *Ann. des Falsif.*, January, February, 1917, xcix., c. 10—14.

† "Korr. Verband. deutsch. Schokoladefab.," 1896, 32.

reports of experimental farms in Canada, and other works dealing with the valuation of waste cacao products, cattle foodstuffs, manures, etc.

Other uses to which cacao husk has been put are for extraction of the fat, preparation of a colouring material, the extraction of theobromine, as soil manure, preparation of a flavour for adding to roasted coffee and its substitutes, or to cheap and inferior cocoa powders and chocolate.

At the present day, with modern efficient machinery, very little cacao-nib dust finds its way into the waste shell receptacle during the process of nibbing, winnowing and grading; consequently, the percentage of fat which commercial cacao husk contains is due, almost entirely, to that present in the shell alone.

The fat-content in cacao husk ranges from 3 per cent. to 6 per cent., and practically all this is capable of extraction by a suitable solvent, such as petroleum ether. The fat so extracted is low in quality, and would be rejected for use in better-class chocolate factories; it does, however, find a market and may, with advantage, be used in the preparation of cosmetics, etc., in which flavour is of little importance.

After the fat has been removed, the residue may be treated with sulphuric acid (3 per cent. solution), boiled, neutralised with baryta, and extracted with chloroform, which removes the caffeine and theobromine, of which the husks contain some 0.6 per cent. Dekker obtained 0.58 per cent. of mixed alkaloids by treatment of the husks with magnesia, boiling with water and extracting with chloroform. The separation of theobromine and caffeine may be accomplished by a further extraction with carbon tetrachloride, or by cautious treatment with caustic soda. In the first case, caffeine is soluble in carbon tetrachloride; in the latter, theobromine is removed, without the caffeine, by the soda.

The husk finds further outlet in chocolate surrogate, or

“suppen powder,” which consists of a small quantity of cacao waste, and sugar, meal, spices and colouring matter in varying quantities. This constitutes a cheap chocolate powder of very inferior quality.

A cacao shell powder of commerce, analysed by the author, showed 9·8 per cent. moisture, 7·2 per cent. ash, 10·2 per cent. fat, 15·6 per cent. albuminoids or proteid matter. Such a powder shows a good value on analysis, but the remaining 57·2 parts was of an indigestible fibrous nature which might reasonably set up irritation in a weak stomach.

“Cocoa tea” of Germany, and “miserables” of Ireland, are independent articles of commerce and consist of cacao husks, in powder or shred, the infusion of which in boiling water is drunk after the manner of tea by the poorer classes of the two countries. As already mentioned, “cocoa teas” made their appearance in England again during the War.

An infusion of cacao husk is not unpalatable and, when concentrated, is used for improving the flavour of cheap cocoas. The preparation of “improvers” from cacao husk forms the subject of many English, French, and German patents.

The composition of cacao husk from beans of different origin does not show important variations; such differences as do occur are attributable to the varying proportions of earth, sand, and similar extraneous matter, found adhering to the outer shell, and to the percentage of moisture which, owing to the different processes of curing and drying undergone, shows considerable variation.

Booth\* has shown some interesting figures which are worthy of reproduction, since they provide information on the alkalinity and cold-water extract, the results for the latter being of importance in the estimation of the cacao

\* N. P. Booth, *The Analyst*, 1909, xxxiv., 144.

present in a sample of cocoa or chocolate.\* These figures should be compared with those given by Arpin, shown in Chapter XIV. of the present work.

TABLE LIX.—*Analyses of Roasted Cacao Husk.*

	Ceylon	African	Para	Guayaquil	Puerto Cabello
Total mineral matter .	6.61	5.63	6.78	8.19	20.82
Soluble mineral matter .	4.78	3.53	4.39	5.25	5.24
Silicious matter .	1.00	1.79	0.72	1.45	8.33
Alkalinity as $K_2O$ .	2.54	2.63	2.80	3.36	1.13
Cold-water extract .	20.70	20.40	18.70	24.60	23.50
Nitrogen .	2.40	2.91	—	2.13	—
Fat .	3.10	3.50	—	5.90	5.68
Fibre .	12.80	15.70	—	12.85	13.83

The average moisture of the shells, shown by Booth, was 4.5 per cent., against 3.0 per cent. for the average of the corresponding nibs, whilst the “cold-water extract” was found to be the same, for all practical purposes, in both nib and shell.

The composition of cacao husk having been now dealt with in the general way, there remains the important consideration of the individual components, which will be taken *seriatim*.

MOISTURE (5 per cent. to 12 per cent. of the raw, 2 per cent. to 8 per cent. of the roasted husk).

The amount of moisture, which any given sample of cacao husk will contain, will depend, firstly, upon the degree to which the particular beans were dried; secondly, to the treatment which they may have suffered during transit or storage; and, thirdly, upon the degree of roasting, and the time of standing before the sample was taken.

There is little need for further discussion on this point, as figures, illustrating the percentage content of moisture in beans fresh from the pod, after fermentation, raw com-

\* Vide Chapter XXX.

mercial, before and after roasting, will be found in this and preceding chapters.

FAT (3.5 per cent. to 8 per cent. of the raw, 4 per cent. to 10 per cent. of the roasted husk).

The fat which is present in the husk is the same as that found in the kernel and is commercially known as "cacao butter," or "cocoa butter," though the latter term should not be used, as confusion will arise between this fat and that obtained from the coconut palm (*Cocos nucifera*).

There is an extremely small proportion of fat in the original fresh husk of the cacao bean, the quantity found in the fermented and dried bean being chiefly due to that absorbed from the kernel.

In Holland, the shells are extracted with ether and benzine, and a low-grade cacao butter is produced, which, however, has more applications in pharmaceutical preparations than for chocolate making, owing to its poor and often unpleasant flavour. By treatment with deodorising carbons, the fat can be produced of quite neutral flavour, which renders it suitable for the foodstuff industries.

Cacao fat is a mixture of glycerides of fatty acids, and contains, besides stearin, palmitin, olein, and laurin, the glyceride of arachidic acid. The occurrence of theobromic acid ( $C_{64}H_{128}O_2$ ) has been mentioned by Kinzett,\* but is disputed by both Graf† and Traub,‡ who were unable to find any fatty acids of higher molecular weight than arachidic.

A very large number of investigators have studied the composition of cacao butter. Hehner and Mitchell§ found

\* C. Kinzett, *Jour. Chem. Soc. Trans.*, 1878, xxiii., 38.

† Graf, *Arch. Pharm.* (3), xxvi., 830.

‡ Traub, *ibid.*, (3) xxi., 19.

§ Hehner and Mitchell, *The Analyst*, 1896, xxi., 321.



40 per cent. of stearic acid ; Farnsteiner \* obtained 59·7 per cent. of saturated acids, 3·12 per cent. of oleic acid, and 6·3 per cent. of other acids. Klimont,† who regards these acids as existing mainly in the form of mixed esters, has isolated the following substances by fractional crystallisation of the fat from acetone :—

(1) A fraction melting at 70° C., having an iodine value = 0. This he regarded as a mixture of tripalmitin and tristearin.

(2) A fraction melting at 31° to 32° C., with a saponification value = 196·4, iodine value = 28·9. This fraction had an empirical formula ( $C_{55}H_{104}O_6$ ), and was judged to be a mixed triglyceride, oleo-palmito-stearin.

(3) A fraction melting at 26° to 27° C., having an iodine value = 31·7, a saponification value = 210, and an empirical formula ( $C_{51}H_{96}O_6$ ). This, Klimont considered to be a mixed glyceride, containing the radicles of myristic, palmitic and oleic acids, and, possibly, of some still lower fatty acids. Triolein was not detected in the fat.

Matthes and Rohdlich,‡ in determining whether cacao butter contained any constituent with a pronounced influence upon the flavour of cacao, succeeded in isolating from the unsaponifiable matter two phytosterols identical, apparently, with sitosterol and stigmasterol, previously isolated by Windhaus from the fat of the Calabar bean. There was also present an oil with a pleasant smell, resembling hyacinth. Fritzweiler§ isolated 6 per cent. of oleodistearin from the fat, which had been removed by Heise's method.

Other contributors to our existing knowledge of the composition of cacao butter, that must be mentioned, are

\* Farnsteiner, *Zeitsch. Nahr. Genussm.*, 1899, ii., 1.

† Klimont, *Monatsh.*, 1902, xxiii., 51 ; 1905, xxvi., 563 ; *Ber. Chem. Ges.*, 1901, xxxiv., 2636.

‡ Matthes and Rohdlich, *Ber.*, 1908, xli., 19.

§ R. Fritzweiler, *Chem. Centr.*, 1902, i., 1113.

Lewkowitsch,\* Strohl,† De Negri and Fabris,‡ Filsinger,§ Wright,|| Rakusin,¶ etc., whose works should be consulted, for further information is required.

According to Schädler, cacao butter has the following elementary composition: Carbon, 75.20 per cent.; hydrogen, 11.90 per cent.; oxygen, 12.90 per cent.

Cacao butter is a yellowish solid, turning gradually white on keeping. It breaks with a smooth fracture, possesses an odour resembling that of cacao and has a smooth and pleasant taste. The fat softens at 26.6° C. and melts between 31.1° and 33.9° C.

The British Pharmacopœia test for its purity is as follows: If 1 grm. be dissolved in 3 ccs. of ether, in a test-tube at 17° C., and the tube placed in water at 0° C., the liquid should neither become turbid nor deposit a granular mass in less than three minutes; and, if the mixture, after congealing, be exposed to a temperature of 15.5° C., it should gradually afford a clear solution (absence of other fats). This is a modification of Björklund's ether test.\*\*

The physical and chemical constants of cacao butter have been estimated by a number of investigators and will be found, classified, in Table LXI. It is, however, desirable to discuss the meaning of these constants, in order to understand their significance.

### *The Specific Gravity.*

The specific gravity, or the "weight of unit volume," of

\* Lewkowitsch, *Jour. Soc. Chem. Ind.*, 1898, xviii., 556, etc.

† A. Strohl, *Zeitch. anal. Chem.*, 1896, xxxv., 166, etc.

‡ De Negri and Fabris, *Ann. Lab. Chim. Centr. delle Gabelli*, 1891, xcii., 258, etc.

§ Filsinger, *Zeitch. anal. Chem.*, 1896, xxxv., 517, etc.

|| Alder Wright, "Oils, Fats and Waxes, and their Manufactured Products," 1903, 529, etc.

¶ M. Rakusin, *Chem. Zeit.*, 1905, xxix., 139, etc.

\*\* Vide Chapter XXIX.

the fat, compared with that of water at the same temperature, is extremely useful in determining the purity or nature of the fat under consideration. In the case of cacao butter this is especially so, as its specific gravity, in the solid state, is higher than the fats with which it is likely to be confounded, or adulterated, whilst, in the liquid and molten state, the specific gravity will be found to be lower than that of its probable adulterants (*vide* Table LXXX.).

In taking the specific gravity of the solid fat, it is necessary to allow the oil to remain solidified for several days, in order that it may assume its normal crystalline form.

It has, moreover, been shown by a number of observers that an expressed fat has a different specific gravity to an extracted fat; it is necessary, therefore, when taking the value, to state by which process the fat has been obtained and to give, also, the age of the sample. Thus Hager gives the normal specific gravity of cacao butter, at 15° C., as 0.95 when fresh, and 0.945 when old. Later values, by the same investigator for fresh butter, are from 0.96 to 0.98 at 15° C. Cacao butter, extracted by solvents, shows a higher specific gravity than the expressed fat, Rammsberger finding values of 0.95 for expressed, and 0.99 for extracted fat.

#### *The Melting-point.*

The melting-point of cacao butter is usually given as 33° C., but figures ranging from 28° to 36° C. are to be found, recorded by various investigators. The differences, found, may usually be attributed to a different source of origin. The melting-point of cacao butter, extracted from beans of various cacaos, has been frequently shown to vary; thus White and Braithwaite and others give the following results:—

TABLE LX.—*Variation of Melting-point of Cacao Butter, with Source of Origin.*

Bean.	Melting-point.		
	I.	II.	III.
Guayaquil Machula . . . . .	34.0	33.75	—
Caracas . . . . .	34.0	33.30	—
Guayaquil Arriba . . . . .	31.5	—	—
Port au Prince . . . . .	33.8	—	—
Puerto Cabello . . . . .	33.0	—	—
Surinam . . . . .	34.0	—	—
Trinidad . . . . .	34.0	32.00	—
Grenada . . . . .	—	33.15	—
Ceylon . . . . .	—	34.05	—
Kauka . . . . .	—	—	32.25
Bahia . . . . .	—	—	33.05
Porto Plata . . . . .	—	—	33.35

- I. Zipperer, "The Manufacture of Chocolate," 1902, 39.  
 II. White and Braithwaite, *Brit. and Col. Druggist*, 1897, xxi.  
 III. *Korr. Verband deutsch. Schokoladefab.*, 1889, v, 65.

Old butter has, moreover, a higher melting-point than fresh, extracted or expressed fat.

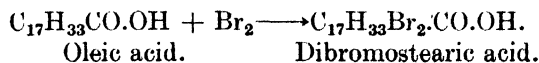
De Negri and Fabris give the melting-point of cacao butter as 28° to 30° C., and the solidifying-point as 21.5° to 23° C.

The determination of the melting-point, by itself, is no criterion of the purity of the cacao butter.

#### *The Bromine and Iodine Absorption Values.*

Organic compounds containing a group of the character CR = CS tend to combine, under suitable conditions, with members of the halogen group, such as iodine and bromine.

By determining the amount of halogen fixed by a given acid or mixture of acids, useful information as to the nature of the fatty acids, present, is obtained. Thus, in a simple case :



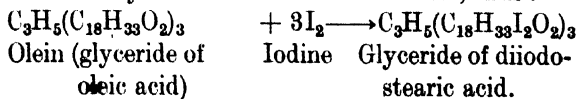
In determining the halogen absorption value, it is more simple to make use of the iodine method, and it is intended here to consider only this quantity, the bromine equivalent being easily found in works treating with oils, fats, etc., or calculated from the iodine figures.

Stearic acid does not absorb iodine, whilst oleic acid takes up 90 per cent. of its weight of the halogen. We have, then, a means of partially discriminating between fats containing varying proportions of olein to stearin. Thus, to take extreme cases, the iodine value for cacao butter is about 35, whilst that for almond oil is about 100, and that of coconut stearin around 4.

Lewkowitsch has shown that the iodine value of cacao butter increases slightly with age, though still not outside the possibilities for normal fat.

The lowest value for iodine absorption by cacao butter, that can be found, is 32.0 by Dietrich, and the highest 41.7 by Strohl, though, again, neither value is much beyond the normal figures of Lewkowitsch, 34.27 to 36.99. Filsinger found that cacao butter from different varieties of beans showed varying figures of iodine absorption, ranging from 35.1 for Porto Plata to 37.1 for Guayaquil Arriba.

The iodine value indicates the amount of iodine per cent. absorbed by the fat and is, consequently, a rough indication of the unsaturated fatty acids of the oleic acid type present. The unsaturated fatty acids directly combine with iodine and bromine to form dibromo- or diiodo-substitution derivatives. The corresponding glyceride derivatives of the unsaturated fatty acids behave in like manner, thus :



The method of bringing about this reaction is discussed in a later chapter, and the value is a very useful indication as to the purity of the fat, under examination.

"Shell butter," or "Dutch HA" cacao butter, has, according to Filsinger, an iodine absorption value of 39 to 40, and is, as a rule, higher than that of the fat extracted from the nib.

*The Saponification or Koettstorfer Value.*

The saponification value is derived by a determination of the number of milligrammes of potassium hydroxide required to saponify 1 gramme of the substance. The various glycerides, of which most of the fats are composed, are constituted so that, on saponification, they break up into glycerol and the alkaline salt of the fatty acid they contain. Owing to the different molecular weights of the various fatty acids, the quantity of alkali that will be required to bring about saponification will vary. The quantity of potassium hydroxide requisite for complete saponification of a fat or glyceride is, therefore, a measure of the molecular weight of the fatty glyceride, or mixture, under consideration, and represents not only the free but the total fatty acids present.

The saponification value of cacao butter ranges from 192 to 202 (Filsinger), and is usually 194 to 195. Dubois has forwarded to the author the saponification values of eleven samples of cacao butter, which range from 192.4 to 195.2. The figures, obtained by various investigators, vary very little, even the ageing of the fat causing but small increase, a fact which is easily understood when it is realised that the saponification value is a measure of the total fatty acids present, which would not be increased by the fat becoming rancid.

*Acid Value.*

The hydrolysis or breaking down of the fats into more simple substances, of which the fatty acids form the principal part, occurs, to a small extent, on the extraction of the fat by means of solvents or expression and always takes place, to a

greater or less degree, after the fat-containing seeds have been detached from the plant. This is brought about by the lipolytic enzymes and ferments of the vegetable kingdom, whose duty it is to break down the fat to form readily assimilable food for the growing plant.

The presence of free fatty acids, in any quantity, is most detrimental to the flavour of a fat, with the exception of olive oil, the flavour of which is supposed to be improved by a small quantity of fatty acids.

In cacao butter, which is less liable to turn rancid, the presence of fatty acids is most objectionable, and it is very easy to recognise a tainted sample.

Lewkowitsch \* has determined the acid value of a number of samples of cacao butter, and, whilst the ordinary fresh fat shows from one to two for a measure of free fatty acid, a sample kept for ten years in a sealed bottle had risen to 4.6. The maximum amount of fatty acids is found in fats extracted by solvents and from beans which have been stored for a considerable time, as, in these cases, the fat has been in contact with the active ferments for the greatest period.

Ballantyne,† however, was unable to trace any connection between rancidity and liberation of fatty acids, though Spaeth ‡ admitted that, in the case of lard, rancidity was attended by a considerable formation of volatile acids.

Rancidity is an oxidation process, brought about by the action of oxygen, light and ferments, and Spaeth observed that, as the oxidation increased, the power of absorbing iodine correspondingly decreased. Fats, thus oxidised, were observed to possess a considerably higher refractive index than the normal fats, a fact which has been attributed to the polymerisation of the unsaturated fatty acids. This

\* J. Lewkowitsch, *Journ. Soc. Chem. Ind.*, 1899, 556.

† Ballantyne, *Journ. Soc. Chem. Ind.*, 1891, x., 29.

‡ Spaeth, *Zeitsch. anal. Chem.*, 1896, xxxv., 471.

has been confirmed by Mjoen,\* though this experimenter was unable to trace the action of bacteria or moulds in the process of oxidation.

The estimation of the chemical and physical constants of the fatty acids, in a manner similar to those of the fats, often affords valuable information as to the nature and purity of the fat, under consideration.

#### *The Reichert-Meissl Value.*

The importance of the Reichert-Meissl value depends upon the fact that various natural oils and fats yield, on saponification, the alkali salts of fatty acids, many of which are volatile in steam. Their estimation constitutes this value.

In the case of butter from cow's milk, the Reichert-Meissl value ranges from 25 to 33, whilst cacao butter never exceeds 1. Lewkowitsch found this value to be the lowest in a cacao butter, kept for ten years in a sealed bottle, and records a figure 0.2. Dubois has forwarded to the author Reichert-Meissl values for eleven samples of cacao butter, varying from 0.10 to 0.61.

\* The actual method of determining these figures will be found in a later chapter.

#### *Refractive Index.*

The power of refracting light, possessed by different oils to a different degree, constitutes a valuable method of discriminating one from another. Suitable instruments have been devised for determining refractive indices, the Zeiss butyro-refractometer, now so extensively used, being, perhaps, the most convenient form.

The refractive index of cacao butter has been recorded by Strohl as 1.4565 to 1.4578 at 40° C., and by Proctor as 1.45

\* Mjoen, *Forsch. Ber.*, 1897, iv., 195.



at 60° C. These figures represent roughly 46° to 48° on the Zeiss butyro-refractometer scale at 40° C.

TABLE LXI.—*Chemical and Physical Constants of Cacao Butter.*

	I.	II.	III.	IV.	V.	VI.
Specific gravity	0.964-0.974 at 15° C.; 0.8577 at 98° C.	—	—	—	0.964-0.974 at 15° C.	—
Melting-point	30°-34° C.	26.6°-33° C.	29°-35° C.	32°-33.6° C.	30°-34° C.	30°-34.5° C.
Iodine value	32.0-42.0	34.27-36.99	32.0-37.0	33.4-37.5	32.0-37.7	34.0-37.5
Saponification value	192.0-195.0	191.8-194.5	193.0-205.0	195.0	—	192.0-202.0
Acid value	—	1.1-4.6	—	—	1.0-2.3	9.24-17.9
Reichert-Meissl value	0.2-0.9	0.38-0.94	—	—	—	—
Refractive index	—	—	Zeiss butyro- refracto- meter value at 40° C. 46-48°	—	—	Zeiss butyro- refracto- meter value at 40° C. 46-47.8°
Acetyl value	—	2.71-2.86	—	—	—	—

The melting-point of the fatty acids, obtained from cacao butter, has been recorded by various investigators as from 47°-49° C. The author has examined one sample which melted at 52° C. The iodine value of the fatty acids has been found to be 39.0 (De Negri and Fabris).

I. Allen's "Commercial Organic Analysis," 1910, vol. II, 71.

II. Lewkowitsch, *Journ. Soc. Chem. Ind.*, 1899, 556; "Chemical Technology and Analysis of Oils, Fats, and Waxes," 1909, etc.

III. Whymper, 1910.

IV. Filsinger, *Korr. Verband. deutsch. Schokoladefab.*, 1889, v, 65, *Zeitsch. anal. Chem.*, 1896, xxxv., 166, etc.

V. Dieterich, in R. Benedikt's "Analysis of Fats and Waxes," etc.

VI. Zipperer, "The Manufacture of Chocolate," 1902, 45.

FIBRE (12 per cent. to 15 per cent. of the raw,  
13 per cent. to 17 per cent. of the roasted husk).

The estimation of fibre in a cocoa powder or chocolate is a guide to the amount of cacao husk, present. Ludwig\* has made a series of analyses of known mixtures of husk and cacao and, by estimating the amount of fibre (by a modified König's method †) in the samples, has given valuable assistance to the analyst desirous of ascertaining the extent of intentional or careless inclusion of cacao husk.

\* Ludwig, *The Analyst*, 1906, 362.

† Vide Chapter XXX.

Six samples of cocoa powder gave 4.98 per cent. to 5.96 per cent. (average 5.60 per cent.) of crude fibre, calculated on fat-free material; the quantity of fat, present in the samples, ranged from 25.05 per cent. to 27.92 per cent., giving an average of 25.78 per cent. The sample of cacao husk contained 3.08 per cent. of fat and 14.47 per cent. of crude fibre.

A mixture of equal parts of the six cocoas was then made, to which increasing proportions of husk were added, with the following results:—

TABLE LXII.—*Crude Fibre in known Mixtures of Cocoa and Husk.*

	Fat.	Crude fibre in fat-free powder.	
		Found. Per cent.	Calculated. Per cent.
Cocoa	25.78	5.50	5.60
" + 5 per cent. husk	24.37	6.03	6.16
" + 10 " " "	24.00	6.39	6.71
" + 20 " " "	21.44	8.06	7.79
" + 40 " " "	16.62	10.18	9.74
" + 60 " " "	12.04	11.72	11.48
" + 80 " " "	8.26	13.28	13.04

The estimation of pentosans in cacao shells has occupied the attention of many observers.

The name of "pentose" is given to that class of compounds in which the alcoholic is joined to the aldehydic function, and their general formula is  $C_5H_{10}O_5$ .

The pentoses, like the hexoses or cane-sugar group, may be found almost everywhere in nature, the gums, arabic gum, cherry-tree gum, etc., being almost entirely composed of pentosans.

R. Adan,\* who has carefully estimated the pentosan-

\* R. Adan, *Bull. Soc. Chim. Belg.*, 1907, xxi., 211.

content of cacao husk, has shown it to contain 7·57 per cent. to 10·53 per cent. of pentosans, as against 1·19 per cent. to 2·19 per cent. for the nibs. P. Welmans \* has given 8·5 per cent. for the husk and 2 per cent. for pure Dutch cocoa as the pentosan-content, whilst Dekker † has published figures ranging from 8·18 per cent. to 9·63 per cent. for the husk, 2·17 per cent. to 2·41 per cent. for the nibs, and 2·56 per cent. for pure Dutch cocoa. Adan ‡ gave some results at the Seventh International Congress of Applied Chemistry held in London, which showed the variations of pentosan-content of husk and nib, of several varieties of cacao. His results are given in Table LXIII.

TABLE LXIII.—*Pentosan-Content of Roasted Nib and Husk.*

	Nib.	Husk (dry).
Arriba . . . . .	1·29	9·97
Port au Prince . . . . .	1·27	7·57
San Thomé . . . . .	1·45	8·49
Caracas . . . . .	1·19	7·78
Bahia . . . . .	1·77	9·45
Soconusco . . . . .	1·21	10·53

Luhrig and Segin § gave 7·59 per cent. to 11·23 per cent. for the pentosans found in husk against 2·51 per cent. to 4·58 per cent. found in the corresponding nibs, and many other observations of this value have been made.

König, || in estimating the amount of cellulose present in cacao husk, found that treatment with sulphuric acid and potash in the usual manner brought about an alteration in the pentosans, so that the results, obtained, did not represent the true amount of cellulose present.

\* P. Welmans, *Zeitsch. öffent. Chem.*, 1901, vii., 491.

† S. Dekker, *Chem. Centr.*, 1902, ii., 1217.

‡ R. Adan, *Internat. Cong. App. Chem.*, 1909, sect. viii., C. 203.

§ H. Luhrig and Segin, *Zeitsch. Nahr. Genussm.*, 1906, xii., 161.

|| König, *ibid.*, 1898, 3

The same author \* recommends the estimation of pentosans by a method with phloroglucine, whilst Tollens † and his pupils have obtained extremely accurate results by estimating pentosans, present, by both volumetric and gravimetric methods, which Adan adopted in his experiments. These methods will be described later.

Fincke ‡ has found in dry fat- and ash-free husks 20.21 per cent. of crude fibre, composed of 9.88 cellulose and 9.92 lignin, against 9.28 per cent. crude fibre, composed of 3.57 cellulose and 5.47 lignin for the dry fat- and ash-free cocoa powder, prepared from the corresponding nibs.

Further discussion of the fibre-, cellulose-, pentosan-, etc., content of cacao will be made when cocoa powders and their

TABLE LXIV.—*Analysis of Mineral Matter from Cacao Husk.*§

	Mara- calbo	Caracas.	Trinidad.	Guaya- quil Machala	Porto Plata.
Ash dried at 100° C.					
I. Insoluble in dilute hydrochloric or nitric acid :					
(a) Volatile above, and dried at, 100° C.	0.11	0.42	0.98	0.31	1.25
(b) Fixed at red heat	1.92	47.71	29.22	37.66	51.51
II. Soluble in dilute hydrochloric or nitric acid :					
(c) Potassium oxide (K <sub>2</sub> O) . . .	31.52	11.81	25.87	23.12	12.17
(d) Sodium oxide (Na <sub>2</sub> O) . . .	4.19	3.30	2.73	1.21	2.78
(e) Calcium oxide (CaO) . . .	10.13	44.46	5.10	3.50	4.40
(f) Magnesium oxide (MgO) . . .	9.55	4.70	5.21	4.84	4.09
(g) Ferric oxide (Fe <sub>2</sub> O <sub>3</sub> ) . . .	0.65	0.93	0.34	0.96	0.46
(h) Aluminium oxide (Al <sub>2</sub> O <sub>3</sub> ) . . .	0.28	1.55	0.71	1.85	1.05
(i) Silicic oxide (SiO <sub>2</sub> ) . . .	1.18	7.98	2.42	4.32	6.78
(k) Phosphoric anhydride (P <sub>2</sub> O <sub>5</sub> ) . . .	9.07	7.63	4.70	7.29	7.24
(l) Sulphuric anhydride (SO <sub>3</sub> ) . . .	3.04	1.48	3.40	1.74	2.01
(m) Chlorine (Cl) . . .	1.01	0.22	1.02	0.26	0.44
(n) Carbonic anhydride (CO <sub>2</sub> ) . . .	25.45	5.40	16.29	11.83	4.25
(o) Water (H <sub>2</sub> O) . . .	2.14	2.50	2.26	1.17	1.66
(p) Oxygen (O) equivalent to chlorine .	0.23	0.05	0.29	0.06	0.10

\* König, "Untersuch. landwirtschaftl. u. gewerbe Stoffe," 1906.

† Tollens, etc., *Zeitsch. angew. Chem.*, 1896, 712, 749, etc.

‡ Fincke, *Zeitsch. Nahr. Genussm.*, 1907, xiii., 265.

§ Vide also Chapter XII., Table XXVII.

possible contamination or adulteration with cacao husk are considered in Chapter XXXII.

ASH (3 per cent. to 9 per cent. of the raw, 3·5 per cent. to 10·5 per cent. of the roasted husk).

The most exhaustive analysis of the ash or mineral matter, obtained from cacao husk, has been made by Bensemann,\* who gives the preceding table.

The most important variations occur in the amount of insoluble mineral matter, fixed at red heat. This would consist chiefly of silica or sand, picked up during the processes of preparation of the beans for market.

Considerable controversy has raged round the mineral matter present in cacao, not so much as to the actual salts present in raw cacao, but with regard to the effect of incineration upon the salts naturally present, and subsequently added, in the preparation of "soluble" cocoa. Baker and Hulton, and Arpin, in papers already quoted here, have considered the alkalinity of the ash especially, and many others have provided complete analyses.

Arpin found the aqueous infusion of shells of cacaos from Trinidad, Madagascar, Carupano, Ibarro, Chuao and Puerto Cabello to be all slightly, or strongly, acid. Working on the ash of these shells, he compiled the following table:—

Raw cacao beans.	Moisture.	Ash.	Alkalinity of the shells. Per cent. ( $K_2CO_3$ ).	
	Per cent.	Per cent.	Raw.	Dried.
Acocra . . . . .	12·28	6·79	5·24	5·97
Bahia . . . . .	13·24	6·60	4·23	4·87
Carupano . . . . .	9·52	7·43	4·69	5·18
Chuao . . . . .	8·60	11·60	4·56	4·99
Madagascar . . . . .	11·93	5·52	4·97	5·64

\* R. Bensemann, *Rep. analyt. Chem.*, 1886, v., 178.

The value of such figures as these has been discussed more fully in those chapters dealing with the alkalinity of cacaos and the detection of added husk to cocoa powders and other cocoa preparations.

Duclaux \* was the first to find small quantities of copper in the ash of cacao, and Galippe,† confirming his results later, found that the greater part of the copper existed in the husk and that, in some samples of inferior chocolate examined, that metal was present in as large a proportion as 0·12 gramme to a kilo of cacao. Gautier, in 1883, had published a book in which he stated that cacao shells contained as much as 0·225 gramme per kilo, but Formenti,‡ in checking these results, obtained 0·014 to 0·040 gramme per kilo in the shells and 0·020 to 0·034 gramme of copper in the nibs.

THEOBROMINE (0·4 per cent. to 2·0 per cent. of the raw or roasted husk).

It has already been pointed out that the theobromine, caffeine and other alkaloids are extracted from cacao husk on the commercial scale. Dekker § was only able to extract 0·5 per cent. of theobromine from the husk, though Eminger || obtained 0·76 per cent. and Wolfram ¶ 0·42 per cent. to 1·11 per cent., the latter figures being obtained from the husk of Caracas beans.

To theobromine and its accompanying alkaloids are due the stimulating properties of cocoa and chocolate, from 1·0 per cent. to 3·0 per cent. being found in the nibs from which the preparations are made. The degree of roasting is an important factor in determining the amount of theobromine to be found in the roasted husks, nibs, and their

\* Duclaux, *Bull. de la Soc. Chim.*, 1872, 33.

† Galippe, *Jour. Pharm. Chim.*, 1883, vii., 506.

‡ C. Formenti, *Zeitsch. Nahr. Genussm.*, 1913, xxv., 149—154.

§ J. Dekker, *Rec. Trav. Chim.*, 1903, xxii., 143.

|| Eminger, *Forsch. Ber. über Lebensmittel*, etc., 1896, iii., 275.

¶ Wolfram, *Zeitsch. anal. Chem.*, 1879, xviii., 346.

preparations, as the alkaloid is volatile, subliming at  $134^{\circ}\text{C}$ . without melting. Knapp, however, on the contrary, has stated that he has found slightly more theobromine in the roasted than in unroasted cacao shells and has attributed the fact to the difficulty with which theobromine sublimes. The same writer has stated that a higher temperature, than that at which cacao is roasted, is required to bring about sublimation, though the accuracy of this statement is open to question.

Woskresensky\* was the first to find theobromine in the seeds of *Theobroma cacao* to the extent of 1.3 per cent. to 4.6 per cent. This early investigator was followed by Glasson, Keller, Rochleder, and Strecker, all of whom obtained higher values than accredited at the present time. Kunze† has made a very complete study of the published analyses and methods of separation of theobromine by Weigmann, Mulder, Wolfram, Zipperer and many others, and has found their methods to be imperfect and their values to be inaccurate.

Fischer‡ was the first to synthesise theobromine which he prepared by heating a lead salt of xanthine ( $\text{C}_5\text{H}_2\text{Pb}_6\text{N}_4\text{O}_2$ ) with methyl iodide; the synthetic product possesses the same physiological and toxic actions as the alkaloid extracted from cacao and has largely replaced the natural product in pharmacy.

Theobromine ( $\text{C}_7\text{H}_8\text{N}_4\text{O}_2$ ) or dimethylxanthine occurs as minute trimetric crystals, slightly soluble in hot water, alcohol and ether, possesses an extremely bitter taste and sublimes, without melting, at  $134^{\circ}\text{C}$ . according to Kunze,† at  $170^{\circ}\text{C}$ . according to Blyth,§ or  $290^{\circ}\text{C}$ . from the results of Schmidt and Pressler.|| Theobromine is slightly soluble in

\* Woskresensky, *Liebig's Annalen*, xli., 125.

† Kunze, *Zeitsch. anal. Chem.*, 1894, xxxiii., 1.

‡ Fischer, *Ber.*, 1907, xv., 453.

§ Blyth, "Foods, their Composition and Analysis," 1909.

|| Schmidt and Pressler, *Liebig's Annalen*, ccxvii., 287.

chloroform and warm amyl alcohol, but practically insoluble in benzol or petroleum ether.

Theobromine forms easily crystallisable salts, the simple neutral salts being decomposed by water into basic salts with loss of acid. An important salt of theobromine is that formed with silver nitrate, quite insoluble in water, possessing a composition, according to Strecker,  $C_7H_7AgNO_2$ .

The alkaloid is precipitated by sodium phospho-tungstate and gold chloride, the former producing a yellow precipitate, whilst the latter yields long needle-shaped crystals.

Theobromine, like caffeine, is closely related to uric acid, and Fischer,\* starting with methyl pseudo-uric acid, succeeded in synthesising the former alkaloid.

The murexide reaction is obtained with both theobromine and caffeine on treatment with chlorine water; the amilic acid, so formed, is rapidly dried down on a watch glass, and a drop of ammonia added, when a violet coloration readily distinguishes these alkaloids from other plant bases which do not belong to the xanthine group.

PROTEID MATTER (12 per cent. to 16 per cent. of the raw, 13 per cent. to 18 per cent. of the roasted husk), and

NITROGEN (2 per cent. to 3 per cent. of the raw or roasted husk).

The nature of the proteid matter, present in the husk of cacao, has been but little investigated, though the large proportion which it contains makes the husk of high food-value, but only for cattle feeding, etc., since the undesirable nature of the other constituents renders it unfit for human consumption. The nitrogen is, however, probably present as globulin, insoluble in water but soluble in solutions of neutral salts.

The shells of beans, fresh from the pod, contain con

\* Fischer, *Berlin chem. Ber.*, 1897, 1839.



siderably larger proportions of proteid matter and nitrogen than the corresponding beans when cured. This is due to the decomposition of the albuminoids, such as globulin, by the agency of the proteolytic enzymes which take a part in the fermentation process, already described, and to the loss of theobromine, which seems to take place, also, during fermentation.\*

Marcker gives from 12.7 per cent. to 14.1 per cent. of albumen, and 4.4 per cent. to 7.1 per cent. of digestible albumen, found in the husks of cacao.

STARCH, CACAO-RED, ETC. (40 per cent. to 55 per cent. of the raw or roasted husk).

The remaining components of husk, constituting some 50 per cent. of the whole, are of little interest or importance, and no figures are obtainable which throw any light upon their actual composition.

This is due to the fact that such analyses of cacao husk as have been made have been carried out with a view to determining its food value—for cattle feeding, etc. A. Smetham † finds 46.01 per cent. of digestible carbohydrates and states that this figure includes all the non-nitrogenous bodies, present in the husk, with the exception of woody fibre and fat. G. Paris and other investigators into the value of cacao husk, for purposes of cattle food, have made indefinite analyses of this portion of the husk components, but they little more than show it to be composed of carbohydrates. It is, however, unnecessary for our purpose to attempt to define it more closely, as the nature of cacao-red and starch likely to be found in the husk, and present in the kernel or nibs to a greater degree, are discussed in the next chapter, where more detailed figures have been collected.

\* Professor Harrison, in Hart's "Cacao," 1900, 105.

† A. Smetham, *Jour. Royal Lancs. Agric. Soc.*, 1900.

Revis and Burnett \* have reported that no *true* starch was found in the cacao shell estimated by a modified Davis and Daish method with taka-diastrase. The shells were from Guayaquil, Trinidad and Grenada beans, whilst the starch found in the nibs of these cacaos amounted to 8·0 per cent., 14·5 per cent., and 12·3 per cent., respectively, when estimated in the same manner (Winton, Silverman and Bailey †).

\* C. Revis and H. R. Burnett, *The Analyst*, 1915, xl., 429.

† Winton, Silverman and Bailey, *Ann. Rep. Conn. Agric. Expt. Stn.*, 1902, 270.

## CHAPTER XXVI

### SURVEY OF THE COMPONENT PARTS OF ROASTED CACAO BEANS—NIBS

#### Nibs.

(80 per cent. to 92 per cent. of the whole bean.)

The amount of nibs obtainable from a cacao depends upon the variety of bean and upon the care with which the husking and the nibbing process has been performed.

Information with regard to both possible variations has been already given, and the amount of nibs, or cacao available for use in the manufacture of cocoa and chocolate, has been shown to range from 80 per cent. to 92 per cent. of the whole bean.

The composition of the kernel or nibs has been studied by many investigators, three of the more recent and important analyses being shown in the following table:—

TABLE LXV.—*Composition of Nib or Kernel (roasted).*\*

	I.	II.	III.
Moisture . . . . .	3.00	5.86	3.11
Fat . . . . .	50.00	50.30	54.37
Starch . . . . .	—	9.97	—
Ash . . . . .	3.07	3.87	3.41
Cellulose . . . . .	—	4.05	—
Pentosans . . . . .	—	1.36	—
Fibre . . . . .	2.80	—	—
Nitrogen . . . . .	2.50	—	—
Cold water extract . . . .	11.60	—	9.67

I. N. P. Booth, *The Analyst*, 1909, xxxiv., 144.

II. R. Adan, *Internat. Cong. App. Chem.*, 1909, viii., C., 203.

III. F. Bordas, *Internat. Cong. App. Chem.*, 1909, viii., C., 188.

\* *Vide* also Chapter XI., Tables XXI., XXII. and XXIII.

# COMPONENTS OF ROASTED CACAO BEANS—NIBS 401

The analyses, shown, are divided by the same three authors into results obtained from different varieties of beans, thus :—

TABLE LXVI.—*Composition of Nibs or Kernels (roasted) from different Varieties of Cacao.*

	Caracas.			Trinidad.		African.		Grenada.		Guayaquil.		Bahia.	
	I.	II.	III.	I.	III.	I.	II.	I.	III.	I.	II.	I.	II.
Moisture . . .	—	7.48	3.27	—	2.90	—	5.71	—	2.86	—	8.52	—	3.71
Fat . . .	—	49.24	52.56	55.70	54.50	50.20	50.20	50.80	55.26	—	50.07	44.40	50.10
Starch . . .	—	9.85	—	—	—	—	13.27	—	—	—	9.10	—	9.61
Ash . . .	3.24	3.92	3.81	2.73	3.30	2.52	3.89	2.00	2.90	3.16	3.89	2.68	3.24
Cellulose . . .	—	4.24	—	—	—	—	4.33	—	—	—	3.70	—	3.93
Pentosans . . .	—	1.19	—	—	—	—	1.45	—	—	—	1.29	—	1.77
Fibre . . .	—	—	—	2.48	—	—	—	2.94	—	—	—	—	—
Nitrogen . . .	—	—	—	2.32	—	1.84	—	2.26	—	—	—	1.98	—
Cold - water extract . . .	—	—	10.87	12.00	9.87	11.80	—	9.80	9.15	11.40	—	9.50	—

More detailed analyses have been made, from time to time, and the three following are typical of the general results obtained :—

TABLE LXVII.—*Analysis of Roasted Cacao Nibs.*

	I.	II.	III.
Moisture . . .	5.23	6.3—8.5	3.7—4.4
Fat . . .	50.44	46.9—52.1	45.3—54.4
Albuminoids . . .	13.26	11.6—21.1	7.4—13.0
Gum . . .	2.17	—	—
Cellulose . . .	6.40	3.3—6.6	—
Alkaloids . . .	0.84	0.3—0.5	—
Cacao-red . . .	2.20	—	—
Ash . . .	2.75	2.9—4.8	2.4—3.9
Astringent matters . . .	6.71	7.2—8.6	26.3—39.4
Cane sugar . . .	—		
Starch . . .	4.20	8.7—12.6	—

I. J. Bell, "The Chemistry of Foods," 1887, 76.

II. Zipperer, "Untersuch. über Kakao, etc.," 56, 57.

III. Heisch, *The Analyst*, i., 142.

The following table, to be found in the records of the Society of Arts, 1874, contains the most important analyses made up to that time:—

TABLE LXVIII.

	I.	II.	III.	IV.	V.	VI.	VII.	VIII.
Fat	53.10	52.00	51.00	50.00	56.00	45.00-49.00	42.67	50.00
Albuminoid substances	18.70	—	—	—	17.00	13.00-18.00	—	—
Albumen	—	20.00	—	20.00	—	—	—	18.00
Fibrin	—	—	—	—	—	—	—	—
Gluten	—	—	20.00	—	—	—	12.21	—
Sugar	—	—	—	—	—	0.00	—	—
Starch	10.01	10.00	—	7.00	—	14.00-18.00	19.03	10.00
Gum	7.75	—	22.00	6.00	—	—	6.40	8.00
Lignin	0.00	—	—	—	22.00	—	—	—
Cellulose	—	2.00	—	—	—	6.08	5.95	—
Woody fibre	—	—	—	4.00	—	—	—	—
Colouring matter	2.01	Traces	—	2.00	—	3.05-5.00	3.96	2.60
Water	5.20	10.00	5.00	5.00	—	5.06-6.30	5.98	6.00
Theobromine	—	2.00	2.00	2.00	1.50	1.02-1.50	0.90	1.50
Salts	—	4.00	—	4.00	—	—	—	—
Ash	—	—	—	—	—	3.05	2.90	3.60
Parts unaccounted for	1.43	—	—	—	3.50	9.14	—	0.30
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

A large number of analyses of the different varieties of beans may be found on referring to the papers mentioned in the footnote, but those given in Table LXVI. may be taken as typical of analyses of commercial value to show the slightly varying composition of several important cacaos.

MOISTURE (6 per cent. to 8 per cent. of the raw,  
4 per cent. to 7.5 per cent. of the roasted kernel).

The variations in water-content of the kernel are due to the same three causes prevailing to alter the percentage of moisture in the husk, already given, namely, degree of drying before shipment, treatment during transit and storage, and temperature and degree of roasting. A large percentage of moisture is, obviously, undesirable in the kernels, as it tends both to deteriorate the flavour of the bean by encouraging fungoid growth and to destroy the keeping property of the cocoa or chocolate, prepared from the damp nibs.

FAT (45 per cent. to 53 per cent. of the raw or roasted kernel).

The percentage of fat in the kernel is a variable quantity, depending slightly upon the variety of bean and upon the degree of roasting.

Harrison has pointed out that, if, during fermentation, germination of the seed is allowed to take place, the fat is one of the first constituents of the kernel to be attacked, after the sugars have been absorbed. This factor may, therefore, play an important part in determining the amount of fat present in any variety, as, in certain districts, germination is allowed to take place before fermentation to a greater or less degree depending upon the procedure of fermentation customary in any district. Davies and McLellan, however, state that the percentage of fat, found, is independent of the locality in which the bean originated.

The following collected figures, obtained from one variety of beans, show some exceedingly contradictory results, but tend to prove that the processes of fermentation and roasting are somewhat overrated as to their importance on final yield of fat, when compared with individual results of other observers.

	Unfer- mented	Mildly fer- mented	Fully fer- mented	Highly fer- mented.	Very high roast.	Very low roast
Fat per cent.	54.68	56.73	57.35	58.23	54.0	52.7

It is our experience that, in the case of a very high roast (above 150° C.), or after a prolonged roasting at a normal temperature, the percentage of fat will be lowered, whilst, the effect of roasting at a reasonable temperature will always raise the fat-content of the nibs, results opposed to the figures given above.\*

The distribution of fat in various parts of the cacao pod and bean has been the subject of several researches.

\* *Vide* Chapter XI.

Harrison has estimated the proportion of the different constituents of cacao in the kernels, cuticles and pulp, and external pod of the fresh cacao fruit, and he has given the values for fat as 30·60 per cent., 0·42 per cent., and 0·14 per cent., respectively, the proportion of moisture being 36·57 per cent., 83·08 per cent., and 84·54 per cent., respectively.

Bordas has found the percentage of fat in different parts of roasted cacao to vary as follows :—

	Per cent.
Cacao nibs (eleven varieties) . . .	50·20—56·14
Cacao dust (from nibbing machine). . .	20·0
Germ . . . . .	3·5
Husk . . . . .	2·0—11·0

The general characteristics of cacao butter have been extensively discussed in the foregoing chapter, where, also, will be found all the important chemical and physical constants for the fat.

STARCH (4 per cent. to 12 per cent. of the raw or roasted kernel) AND OTHER CARBOHYDRATES (8 per cent. to 13 per cent. of the raw or roasted kernel).

The starch, present in cacao, is similar in composition to that found in other vegetable seeds. It is readily gelatinised by boiling water, is turned blue by iodine and is converted, by the action of dilute mineral acids and diastase, into glucose.

The granules of cacao starch are very minute, and the concentric rings, showing their composite structure, can only be detected with a high-power microscope.

Bordas has made careful determinations of the quantity of starch to be found in different cacaos and has stated that the amount is practically constant for all commercial varieties. These results are as follow :—

Harrison, in Hart's "Cacao," 1900, 90.

TABLE LXIX.—*Starch in different Varieties of Cacao.*

	Starch in 100 parts of cacao.	Starch calculated for 100 parts of cacao insoluble in water.
Sancheize . . . .	9.60	28.90
Bahia . . . . .	9.00	27.60
Haiti . . . . .	8.50	26.10
St. Lucia . . . .	9.29	28.49
Trinidad . . . .	9.60	29.77
Grenada . . . . .	9.35	28.56
Maragnan . . . .	8.88	27.66
Carupano . . . .	9.93	30.30
Porto Plata . . .	10.00	30.50
Caracas . . . . .	9.93	29.81
Guadeloupe . . .	8.66	26.00
Mean . . . . .	9.34	28.50

Maurenbrecker and Tollens \* have made a close study of the carbohydrates occurring in cacao and have succeeded in detecting 1.-arabinose, d.-galactose and glucose (from hydrolysis of the starch), but no xylose was found. The same authors estimated the amount of pentosans, already discussed in a preceding chapter, and found them to be present to the extent of 2.25 per cent. in the nibs or kernels, and 9.02 per cent. to 9.09 per cent. in the husks. These figures are in close agreement with those obtained by Dekker,† who found 2.17 per cent. to 2.41 per cent. of pentosans in the kernel, and 8.18 per cent. to 9.63 per cent. in the husks.

Adan ‡ obtained figures equivalent to 1.53 per cent. of pentosans for the nibs, and 9.96 per cent. for the dry husks.

More recently, Revis and Burnett § have made some very careful estimations of starch. From their results, they have

\* A. D. Maurenbrecker and B. Tollens, *Ber.*, 1906, xxxix., 3576.

† Dekker, *Chem. Centr.*, 1902, ii., 1218.

‡ R. Adan, *Bull. Soc. Chim. Belg.*, 1907, xxi., 211, and *Internat. Cong. App. Chem.*, 1909, viii. C, 203.

§ C. Revis and H. R. Burnett, *The Analyst*, 1915, xl., 429.



stated: "It is evident that cacao shell contains no *true* starch, and it is probable that this is the first time that it has been demonstrated analytically." In the nibs, the same experimenters found from 8 per cent. to 14.5 per cent. of starch, calculated on the fat-free cocoa.

Nibs	Percentage of starch in fat-free nibs.
Accra (low grade cacao)	12.8
Guayaquil	8.0
Trinidad	14.5
Grenada	12.3
San Thomé	12.4

FIBRE (2 per cent. to 5 per cent. of the raw or roasted kernel).

The amount of fibre, found in the nibs, is entirely dependent upon the care with which the husk or fibrous matter is separated from the inner kernel. It has been pointed out by Tollens, König and others that the estimation of cellulose is not a true guide to the quantity of fibre present, as the existence of pentosans hinders the correct determination.

The fibrous wall of the cacao bean is composed of pentosans, embedded in many other matters, such as cacao-red, mineral matter, etc., and it is in the estimation of the pentosans that addition of husk to cocoa preparations is best detected. If properly freed from husk, the nibs should not contain more than 4.5 per cent. of crude fibre, though Filsinger\* obtained values ranging from 2.8 per cent. to 5.4 per cent. for different varieties of beans, by estimating the crude fibre by a method described by König. Such a figure for crude fibre would approximately equal 2.5 per cent. of pentosans, constituting a little more than 50 per cent. of the total crude fibre found.

Welmans† obtained an average of 13.3 per cent. of husk from raw, and 12.4 per cent. from roasted nibs. His figures

\* Filsinger, *Zeitsch. öffent. Chem.*, 1900, 223.

† P. Welmans, *Zeitsch. öffent. Chem.*, 1901, vii., 491.

are, obviously, too high for a well-husked kernel, and it is to be assumed that they comprise the ash, fat and other components of the husk, besides crude fibre.

Bordas, in his communication to the International Congress of Applied Chemistry, 1909, has shown the amount of cellulose present in various cacaos. He has pointed out the value of cellulose estimation, on the ground that any addition of matter containing woody fibre, such as husks, cacao waste, germs, etc., will increase the proportion of cellulose and diminish the starch-content of the kernels or of the cacao mass, prepared from them. His results will be found in Chapter XII., Table XXVI.

Ash (3 per cent. to 4 per cent. of the raw or roasted kernel).

The percentage of ash in cacao beans has been shown by the investigations of a number of observers to vary considerably. This is usually the case in the mineral matter obtained from foodstuffs of vegetable origin.

Bensemann\* has made careful analyses of the ash, separated from different varieties of cacao, and his figures for

TABLE LXX.—*Analyses of Mineral Matter from Cacao Nibs.*

	Caracas.		Trinidad.	
	I.	II.	I.	II.
Potassium oxide ( $K_2O$ ) . . . .	33.64	32.50	30.85	32.28
Sodium oxide ( $Na_2O$ ) . . . .	0.77	2.50	1.96	1.90
Calcium oxide ( $CaO$ ) . . . .	5.03	4.30	4.64	3.54
Magnesium oxide ( $MgO$ ) . . . .	15.15	13.00	16.06	15.43
Ferric oxide ( $Fe_2O_3$ ) . . . .	0.22	1.82	0.49	0.74
Aluminum oxide ( $Al_2O_3$ ) . . . .	0.33	7.00	0.17	7.00
Silicic oxide ( $SiO_2$ ) . . . .	0.21	25.60	28.62	28.38
Phosphoric anhydride ( $P_2O_5$ ) . . . .	29.30	3.10	3.96	3.80
Sulphuric anhydride ( $SO_3$ ) . . . .	2.74	0.34	0.43	0.40
Chlorine (Cl) . . . .	0.34	7.98	8.95	5.53
Carbonic anhydride ( $CO_2$ ) . . . .	8.44	—	—	—
Water ( $H_2O$ ) . . . .	1.98	—	—	—

I R. Bensemann, *Rep. analyt. Chem.*, 1885, v., 178.

II. Dr. Bordas, *Internat. Cong. App. Chem.*, 1909, viii. C, 188

\* R. Bensemann, *Rep. analyt. Chem.*, 1885, v., 178.

the mineral matter from Caracas and Trinidad cacaos are shown above, compared against those obtained by Bordas, whose work has already been quoted.

Bordas' figures for silicic oxide include sand, and those for carbonic anhydride are estimated by difference.

It will be seen that the most important component of cacao ash is potassium oxide ( $K_2O$ ), which amounts to about one-third of the total mineral matter. The potassium is probably present in the original bean as salts of the plant acids and as carbonate.

The lime, or calcium oxide, is present as oxalate to the extent of 0.2 per cent. of the nib, other alkaline oxalates being found by Girard \* to amount to 0.15 per cent. of the roasted kernel or nib of Trinidad cacao.

The amount of potassium salts, present, is of interest when the contentions that the addition of alkali, such as potassium hydrate or carbonate, to cocoa or chocolate is injurious to health are considered.

Luhrig and his co-workers † have estimated the alkalinity of the aqueous extract of cacao beans and, from the average of twenty-eight samples, obtained a value of 0.99 per cent. of alkali, as  $K_2CO_3$ , on the fat-free cacao. One sample of cocoa powder yielded as much as 2.11 per cent. of alkali, soluble in water, calculated as  $K_2CO_3$  on dry cocoa powder, after 80 per cent. of fat had been extracted. The same author has pointed out that, if the amount of alkali, calculated as potassium carbonate ( $K_2CO_3$ ), exceeds 3 per cent., the addition of alkali is certain. These figures are considerably in excess of those obtained by Booth ‡, already given.

Arpin, in a paper already quoted, has given the alkalinity of some raw nibs as varying between 2.92 and 3.59 per cent., calculated as  $K_2CO_3$  on the dry fat-free cacao.

\* C. Girard, *Internat. Cong. App. Chem.*, 1909, viii. C, 179.

† Luhrig, etc., *Zeitsch. Nahr. Genussm.*, 1905, ix., 257.

‡ N. P. Booth, *The Analyst*, 1909, xxxiv., 143.

PROTEID MATTER (10 per cent. to 17 per cent. of the raw or roasted kernel), and NITROGEN (1·5 per cent. to 2·5 per cent. of the raw or roasted kernel).

Leffman and Beam \* have recorded some results, obtained by Stutzer, on the nitrogenous constituents of cacao.

Stutzer † found that the nitrogenous components were of three types :—

(1) Non-proteids, not precipitated by  $\text{Cu}(\text{OH})_2$ , such as theobromine, caffeine and amido compounds.

(2) Digestible albumen, insoluble in water in the presence of  $\text{Cu}(\text{OH})_2$ , but soluble when treated with acid, gastric juice and alkaline pancreatic extract.

(3) Insoluble and indigestible nitrogenous matter.

The analyses of the nitrogenous constituents are given as follows :—

	I. Per cent.	II. Per cent.	III. Per cent.
Nitrogen as soluble compounds (alkaloidal)	31·43	26·95	29·79
Nitrogen as digestible albumen	33·34	40·61	22·62
Nitrogen as indigestible matter	33·33	32·44	47·83

The three nitrogen-bearing constituents were present, according to this author, in approximately equal proportions. The results obtained by other observers do not agree with Stutzer's figures : thus, Bell gives albuminoids (proteid matter, or digestible and indigestible proteid) as 13·26 per cent., and alkaloids as 0·84 per cent. of the kernel. Zipperer has found 11·6 per cent. to 21·1 per cent. of albuminoids, and 0·3 per cent. to 0·5 per cent. of alkaloids in the kernel. The results obtained by Bell and Zipperer are out of date and cannot be relied upon.

The percentages and quality of the total nitrogen-containing components of cacao are of little commercial import-

\* Leffman and Beam, " Food Analysis," 1901, 275—282.

† Stutzer, *Zeitsch. phys. Chem.*, ii., 207.

ance, however, and Stutzer's figures are also obviously open to criticism, owing to the large error which occurs on multiplying the small quantity of nitrogen, actually found for each type, to read as percentage of total nitrogen.

The amount of nitrogen multiplied by 6.25 gives, approximately, the total albuminoid matter found in cacao.

THEOBROMINE (0.9 per cent. to 3 per cent. of the raw or roasted kernel) and CAFFEINE (0.05 per cent. to 0.36 per cent. of the raw or roasted kernel).

The general chemical and physical characteristics of the alkaloids, present in cacao, have been given in a previous chapter.

The amount of these alkaloids, found in the nibs, varies very considerably, both with the variety of the bean and with the care of the observer. Great differences are to be found between the alkaloid estimation of early and recent investigators; thus Zipperer finds 0.3 per cent. to 0.5 per cent. of

TABLE LXXI.—*Theobromine in different varieties of Cacao.*

	I.	II.	III.	IV.	V.
Caracas . . . . .	1.43	1.63	0.50	1.13	1.38
Guayaquil . . . . .	0.9—1.2	1.63	0.30	0.86	—
Domingo . . . . .	1.98	1.66	—	—	—
Bahia . . . . .	2.04	1.64	—	1.08	—
Puerto Cabello . . . . .	1.02	1.46	0.52	—	—
Maracaibo . . . . .	1.84	—	—	1.03	—
Ceylon . . . . .	2.06	—	—	—	—
Java . . . . .	2.34	—	—	1.16	—
Trinidad . . . . .	1.98	—	0.42	0.85	1.44
Para . . . . .	1.08	—	—	—	1.28
Grenada . . . . .	1.90	—	—	0.75	1.60
Surinam . . . . .	1.83	—	0.54	0.93	—
Cameroon . . . . .	1.83	—	—	—	—
San Thomé . . . . .	2.09	—	—	—	—
Haiti . . . . .	2.07	—	—	—	—

I. A. Eminger, *Apoth. Zeit.*, 1896, 716.

II. Wolfram, *Zeitsch. anal. Chem.*, xviii., 346.

III. Zipperer, "Untersuch. über Kakao, etc.," 1886, 56.

IV. Ridenour, *Amer. Journ. Pharm.*, 1895, lxvii., 202.

V. V. L. Maupy, *Journ. Pharm. Chim.*, 1897 [6], v., 329.

total alkaloids in the roasted nibs of cacao, and Eminger 1.0 per cent. to 2.3 per cent. of theobromine and 0.05 per cent. to 0.36 per cent. of caffeine.

Kreutz \* has pointed out a possible explanation of these discrepancies and has stated that theobromine exists in the bean partly as the free alkaloid and partly in the form of a glucoside which can only be separated after treatment with an acid. The same author has found from 1.5 per cent. to 2.4 per cent. of free, and from 1.6 per cent. to 2.8 per cent. of combined alkaloids in the kernels of cacao.

Table LXXI. shows the results obtained by several other observers.

CACAO-RED (2 per cent. to 5 per cent. of the raw or roasted kernel).

The colouring matter of cacao is produced during the oxidation of the beans.

When fresh, the Criollo variety of bean is white, or nearly so, the Calabacillo variety being, often, of a deep purplish tint, whilst the Forastero cacao, occupying the intermediate position, shows only a faintly coloured kernel.

Harrison has shown that there is a loss of cacao-red in all three varieties during fermentation, a total loss of 0.27 per cent. occurring in a mixture of Calabacillo and Forastero beans.

The formation of cacao-red is of peculiar importance to the manufacturer, who looks to this agency for the bright chocolate-colour of his goods. The variety of bean, selected, and careful roasting are the most important factors in obtaining a satisfactory colour for cocoa or chocolate. Schweitzer † has made some investigations into the formation of cacao-red in the bean and has compared it to the production of colouring matter in the oak, kola nut and

\* A. Kreutz, *Zeitsch. Nahr. Genussm.*, 1908, xvi., 579.

† C. Schweitzer, *Pharm. Zeit.*, 1898, 389.

quinine, tracing it to the decomposition of a glucoside, "cacaonin," during fermentation, when theobromine, caffeine and glucose are also produced.

Hilger,\* also, has closely followed the production of cacao-red, during the fermentation of the bean, and has concluded that the colouring matter, isolated in the ordinary way, is a mixture of non-nitrogenous cacao-red and some glucoside.

True cacao-red can be isolated by treating the roasted beans first with petroleum ether, to remove the fat and part of the free theobromine, then with water, to extract the remainder of the theobromine, caffeine, sugars and salts, and, finally, with alcohol, to extract the cacao-red. A substance, having a formula  $C_{17}H_{12}(OH)_{10}$ , is obtained in this way, closely akin to tannin which it resembles in yielding formic acid, acetic acid and pyrocatechin, on treatment with potassium or sodium hydrate.

Blyth † states that the fat-free cacao is only partially deprived of its cacao-red by solvents, unless a mineral acid has been previously added. After the addition of a few cubic centimetres of hydrochloric acid, the red colouring matter is dissolved, with ease, by amyl or ethyl alcohol. Cacao-red, so produced, is insoluble in ether or petroleum ether, but slightly soluble in carbon bisulphide. It is a sensitive reagent to acid and alkali, mineral acids causing a red coloration with violet shimmer, alkalis usually striking a dirty green. The aqueous solution of cacao-red, of this form, is bitter and astringent and forms precipitates with salts of iron, copper and silver.

A more recent contribution to our knowledge of cacao-red has been given by Reutter‡ who, first of all, extracted fat-free cacao with hot, dilute methyl alcohol. From the violet-red

\* Hilger, *Apoth. Zeit.*, 1892, 469; also *Deutsch. Viert. öffentl. Gesund.*, 1893, 3.

† A. W. Blyth, "Foods, their Composition and Analysis," 1909, 363—375.

‡ L. Reutter, *Compt. Rend.*, 1913, civi., 1842—1844.

alcohol solution, he obtained white microscopic crystals of a new substance, named by him "cacaorine" ( $C_{16}H_{20}O_6N_8$ ), which had a melting point  $184-185^{\circ}C$ . This compound is, evidently, similar to Schweitzer's "cacaonin," since an aqueous solution, after hydrolysis, yielded theobromine and a small quantity of a brownish red precipitate. By concentrating, *in vacuo*, the mother liquor from which the "cacaorine" had been obtained, Reutter isolated, as violet-red spangles, a colouring matter which he called "cacao-red" ( $C_{40}H_{60}O_{27}N$ ). "Cacao-red," as prepared, was soluble in alcohol and water, and its aqueous solution was coloured brownish yellow by dilute alkalis, bright red by dilute acids, blood red by picric acid, green by copper acetate, and violet by ammoniacal zinc acetate. After hydrolysis, a brownish precipitate, "cacao-brown" ( $C_{76}H_{78}O_{34}N$ ), was formed, and the aqueous solutions became slightly dextrorotatory.

For further particulars of cacao-red, the reader is referred to Chapter VI., where a more detailed account of the development of colour in cacao, during fermentation, has been discussed.

#### ORGANIC ACIDS.

TARTARIC ACID (free), about 0.15 per cent.	} of the raw kernel.
ACETIC ACID (free), about 0.25 per cent.	
OXALIC ACID (total), about 0.35 per cent..	

Harrison, who has made a complete study of the changes occurring in cacao during fermentation, has found that tartaric acid is present in larger proportions in the fermented and cured beans than in those simply dried. The same remarks apply to the quantity of free acetic acid present, though, in this case, the dried beans were found to contain none, or only negligible quantities,



whereas the fermented and cured beans contained as much as 0.7 per cent.

The amount of free tartaric acid, contained by the fresh beans (0.25 per cent.), diminished to 0.2 per cent. during the process of fermentation and to 0.12 per cent. during simple drying.

The free acetic acid, which was *nil* in the fresh beans, increased to 0.33 per cent., during fermentation and curing, and was again *nil* after the fresh beans had been simply dried.

The quantities of these two acids in the free state were found by the same author to vary slightly with the three varieties of cacao, Criollo, Forastero and Calabacillo. The differences are, however, small and of no commercial importance.

The acetic acid can be detected in beans that have been fermented by the vinegary smell which, during the process of roasting, it is so desirable to remove.

The presence of oxalic acid in the vegetable kingdom has been recognised since the commencement of the nineteenth century—Fourcroy and Vauquelin \* have stated that this acid is to be found in almost every plant. The method by which their approximate results were obtained was by means of the microscope. It was as recently as 1900 that the first reliable method for the estimation of oxalic acid in cacao was published, the outcome of continuous research on the parts of Neubauer,† Schultzen,‡ Salkowski,§ Albahary || and others. The method was based on the fact that 100 grammes of ether dissolve 1.27 grammes of oxalic acid, and that the solubility of the acid could be increased by the addition of alcohol.

\* Fourcroy and Vauquelin, *Jour. Phys.*, lxxviii., 429.

† Neubauer, *Zeitch. anal. Chem.*, 1868, ii., 499, and vii., 225.

‡ Schultzen, "Reichert und Dubois Reymond's Arch.," 1868, 718.

§ E. Salkowski, *Zeitch. phys. Chem.*, x., 120.

|| J. M. Albahary, *Compt. Rend.*, cxxvi., 1681, etc.

Gautier gives figures, found by Esbach, for the oxalic acid content of several vegetable foodstuffs, thus :—

	Per cent
Cocoa powder . . . . .	0.45
Chocolate . . . . .	0.09
Black tea . . . . .	0.38
Infusion of tea . . . . .	0.21
Coffee . . . . .	0.01
Rhubarb . . . . .	0.25

Girard has recently made some very careful estimations of the amount of oxalic acid present in cacao beans. The following table shows his results, obtained from Trinidad cacao :—

TABLE LXXII.—*Oxalic Acid present in Trinidad Cacao.*

	Roasted beans	Raw beans	Roasted nibs.	Raw nibs	Roasted husk.	Raw husk	Roasted germ.
Calcium oxalate . . . . .	0.195	0.191	0.221	0.216	0.121	0.119	0.240
Oxalic acid due to calcium oxalate . . . . .	0.137	0.163	0.155	0.152	0.198	0.084	0.169
Oxalic acid due to alkaline oxalate . . . . .	0.172	0.134	0.160	0.158	0.085	0.189	0.150
Total oxalic acid . . . . .	0.309	0.297	0.315	0.310	0.283	0.273	0.319
Water . . . . .	3.76	5.44	3.56	4.64	9.04	11.52	3.50
Total oxalic acid per 100 parts of dry cacao . . . . .	0.321	0.314	0.326	0.325	0.301	0.301	0.321

From an examination of the oxalates, found, the same author concludes that :—

(1) Trinidad cacao contains oxalic acid in the same degree as sorrel, spinach and rhubarb.

(2) Oxalic acid is to be found in approximately the same proportion in the beans, nibs, husk and germ.

(3) Roasting has little or no effect on the oxalic acid-content of the products.

(4) A considerable proportion of the oxalic acid exists in the state of alkaline oxalates.

## ESSENTIAL OIL.

Reference has already been made to the work of Bainbridge and Davies on the development of essential oil, during the fermentation of cacaos. These workers took 2,000 kilos of cacao nibs which they steam-distilled. From the distillate, 24 ccs. of a purified oil were obtained, which consisted of a mixture of esters with the true essential oil. The true oil was found to consist chiefly of d.-linalool. Octoic acid and other fatty acids, probably derived from the decomposition of the cacao butter, were also isolated, as well as a small proportion of a stable nitrogenous compound which was not identified.

## • CHAPTER XXVII

### COCOA POWDERS AND CHOCOLATES, AND THEIR COMPONENT PARTS

•  
THE component parts of cocoa powders should be the same as those of the nibs, already dealt with in the last chapter. During the preparation of cocoa powder, however, a certain quantity of fat is extracted, usually about 30 per cent. of the total fat present, and, consequently, the other components are found in the powders in correspondingly larger proportions.

Again, the cocoas may have been treated with alkalis, which, if carbonate or hydrate of sodium and potassium, will cause an increase in the amount and in the alkalinity of the ash, found.

Chocolate powders, on the other hand, usually consist of cocoa powder or cacao mass mixed with sugar, and, frequently also, with starch of potato, sago, arrowroot, etc.

Plain chocolates, as distinguished from fancy goods consisting of fruit jelly, nougat, almonds, sugar fondant and other centres covered with a thin coating of chocolate, should consist of cacao mass, sugar, and added cacao butter only.

Milk chocolates should contain the solids of fresh full-cream milk, besides those ingredients which go to make up plain eating-chocolates.

Nut chocolates, coffee chocolates, etc., should consist of plain chocolate with the addition of the ingredients which give the name to the speciality.

**Cocoa Powders.**

In Chapter XIV. will be found analyses of many varieties of commercial cocoa powders. It is unnecessary, therefore, to reproduce them here, though it is desirable that some average analyses should be given for easy reference.

In the following table, figures for four typical varieties of cocoas are shown : (1) ordinary cocoa powder from expressed cacao nibs ; (2) cocoa powder treated with alkali ; (3) cocoa powder treated with ammonia ; (4) adulterated cocoa powder (given by Booth) ; (5) "Cocoa teas" (selected from Baker and Hulton's results, and approximated).

Where it has been found possible, one author's results have been supplemented with those obtained by another observer, who gives closely agreeing figures for the main components.

TABLE LXXIII.—*Analyses of Cocoa Powders.*

	I.	II.	III.	IV.	V.
Moisture . . . . .	4.33	4.53	6.56	2.40	10.00
Total ash . . . . .	4.28	8.19	5.18	2.75	9.00
Alkalinity as $K_2O$ . . . .	1.00	3.68	—	—	4.00
Fat . . . . .	30.95	29.78	27.34	0.86	8.00
Extractive soluble in water .	18.50	17.45	—	—	20.00
Theobromine . . . . .	1.36	0.60	1.98	—	—
Starch . . . . .	16.07	21.26	—	—	—
Albumen . . . . .	12.78	17.03	10.50	—	14.00
Fibre . . . . .	3.89	4.38	4.00	10.90	15.00
Foreign starch . . . . .	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	30.00	—
Character of starch . . . .	—	—	—	Sago.	—
Sugar . . . . .	<i>Nil.</i>	<i>Nil.</i>	<i>Nil.</i>	38.00	—

MOISTURE (2.25 per cent. to 5.0 per cent. of cocoa powder).

A good cocoa powder should never contain more than 5 per cent. of moisture, as an amount exceeding this limit tends to "ball" the powder and to render it liable to the attack of mildew, which would seriously injure the flavour.

ASH (8 per cent. to 5 per cent. of cocoa powder, 5 per cent. to 11 per cent. of "soluble" cocoa powder).

In Chapter XIV.; Table XXXVIII., will be found analyses of the ash, obtained by incinerating some cocoa powders of commerce, carried out by Girard.

Bensemann and Bordas have also made careful analyses of the ash, obtained from different varieties of cacao beans.

The following table shows the collected results of the different authors :—

TABLE LXXIV.—*Analyses of Ash of Cacao Mass and Cocoa Powders.\**

	I.	II.	III.
Potassium oxide ( $K_2O$ ) . . . . .	32.25	29.53	22.97
Sodium oxide ( $Na_2O$ ) . . . . .	2.17	2.44	22.32
Calcium oxide ( $CaO$ ) . . . . .	3.96	3.39	1.25
Magnesium oxide ( $MgO$ ) . . . . .	16.14	16.57	10.33
Ferric oxide ( $Fe_2O_3$ ) . . . . .	0.36	0.49	0.83
Aluminium oxide ( $Al_2O_3$ ) . . . . .	0.33	0.08	0.15
Silica ( $SiO_2$ ) . . . . .	0.19	1.67	0.77
Phosphoric anhydride ( $P_2O_5$ ) . . . . .	31.59	32.41	24.13
Sulphuric anhydride ( $SO_3$ ) . . . . .	3.07	2.58	1.84
Chlorine (Cl) . . . . .	0.29	0.61	0.96
Carbonic anhydride ( $CO_2$ ) . . . . .	6.80	10.35	14.67
Water ( $H_2O$ ) . . . . .	1.94	Nil.	Nil.

I. Ash from cacao mass, by R. Bensemann, *Rep. analyt. Chem.*, 1885, v., 178.

II. Ash from Menier cocoa powder, by C. Girard, *Internat. Cong. App. Chem.*, 1909, viii, C, 186.

III. Ash from Van Houten's cocoa powder, by C. Girard, *ibid.* \*

From these figures, it will be seen that the most important differences arise in the values of sodium oxide and carbonic anhydride in Samples I. and III. In the latter case, it is certain that sodium hydrate or carbonate was used for rendering the cacao "soluble," the ash, in either case, being capable of estimation as sodium oxide and carbonic anhydride, due to the absorption of the latter from the air by the alkaline hydrate.

\* Vide also Chapter XII., Table XXVII.

Bordas,\* elsewhere, has pointed out that the quantity of added alkali, present in imported cocoas, should be regulated, Belgium, Italy and Switzerland already prohibiting the sale of cocoa powders containing more than 3 per cent. of added alkaline carbonate. Reference has already been made to the regulations existing in France in Chapter XIV.

The same author points out that natural cacao mass contains from 2.46 per cent. to 3.05 per cent. of alkali, calculated as potassium oxide on the fat-free substance, whilst samples of "soluble" cocoa, examined, have shown from 4.82 per cent. to 6.41 per cent. of added alkali, calculated on the same basis.

Stutzer † has estimated the soluble constituents of many samples of cocoa and has stated that there is nothing to be gained by the addition of alkaline carbonates. With this opinion we do not agree. ‡

Matthes, § in conjunction with Rohdlich, has formed the opinion that the estimation of silica ( $\text{SiO}_2$ ), previously suggested by the former author, is no indication of the amount of husk added to a cocoa powder, as they found, in twenty samples of cocoa examined, figures ranging from 0.02 per cent. to 0.88 per cent. of silica. Except in extreme cases of adulteration with husk, this is<sup>d</sup> true, Bensemann finding from 1.2 per cent. to 8.0 per cent. of silica in the husk and 0.2 per cent. in the nibs, whilst Bordas has given from 0.5 per cent. to 7.3 per cent. of silica and sand in the nibs and 19.10 per cent. in the husk.

The discussions raging round the ash content and its alkalinity have been considered elsewhere, but it is desired to refer the reader again to the results obtained by Arpin.||

\* F. Bordas, *Ann. des Falsif.*, 1910, iii., 61.

† A. Stutzer, *Zeitsch. angew. Chem.*, 1892, 510.

‡ Vide Chapters XIII., XXIII. and XXIV.

§ Matthes and Rohdlich, *Zeitsch. öffent. Chem.*, 1908, xiv., 166.

|| Arpin, *Ann. des Falsif.*, January, February, 1917, xcix., c.

Starting with nibs of different cacaos, this experimenter examined cocoa powders, husk and germ with regard to the alkalinity of their ash, in order to ascertain whether the French decree of 1910 could be upheld.

The following analyses were obtained from unroasted shelled nibs :—

	Trinidad.	Mada- gascar.	Caru- pano	Barra.	Chuao.	Puerto Cabello.
Moisture, per cent. . . . .	6.36	6.37	7.23	7.21	7.27	7.91
Fat, per cent. . . . .	47.60	48.40	42.70	43.80	43.80	46.90
Ash, per cent. . . . .	2.95	3.16	2.90	2.96	3.63	3.16
Alkalinity (as $K_2CO_3$ per cent. of dry defatted cocoa) . . . . .	3.10	3.53	2.92	3.10	3.59	2.96

The unroasted husk of some of these beans showed the following analysis :—

	Accra	Bahia	Carupano	Chuao.	Mada- gascar.
Moisture, per cent. . . . .	12.28	13.24	9.52	8.60	11.93
Ash, per cent. . . . .	6.79	6.60	7.43	11.60	5.52
Alkalinity (as $K_2CO_3$ on the raw husk) . . . . .	5.24	4.23	4.69	4.56	4.7
Alkalinity (as $K_2CO_3$ on the dried husk) . . . . .	5.97	4.87	5.18	4.99	5.64

Roasted germ of Trinidad cacao showed, on analysis :—

Moisture, per cent. . . . .	4.44
Fat . . . . .	4.44
Ash . . . . .	6.25
Alkalinity of ash, as $K_2CO_3$ per cent. of dry and defatted germ . . . . .	4.13

Three cocoa powders, purchased in the open market by the French "Service for the Suppression of Fraud," were analysed and were found to fall outside the limit of alkalinity for cocoa powders "guaranteed pure," a term to be applied only to those powders the percentage alkalinity of



the 65 samples of Dutch cocoas, examined, 49 contained less than 20 per cent., 3 less than 25 per cent., and 13 samples over 25 per cent. of fat; all the Swiss samples contained over 25 per cent. of fat.

EXTRACTIVE SOLUBLE IN WATER (13.5 per cent. to 18.5 per cent. of cocoa powder).

The cold-water extract of the nib or kernel of cacao ranges from 9.5 per cent. to 12.5 per cent., whilst the figure for fat-free cacao amounts to approximately 24 per cent. Florence Yaple,\* however, gives considerably lower figures, which we are unable to reconcile with those found by more recent observers.

The soluble extractive, which will include sugars, gums, soluble starch, soluble mineral matter, etc., is a useful indication to the possible adulteration of a powder with sugar and alkali, the addition of either tending to increase the figure for soluble extractive.

The alkalinity of the extractive, soluble in water, will be a guide to the extent of addition of alkali.

The estimation of soluble extractive is of greatest importance in the analysis of chocolate or chocolate powders, as well as in cocoa.

THEOBROMINE (0.70 per cent. to 2.70 per cent. of cocoa powder).

The quantity of theobromine, found in a cocoa powder, will depend largely upon the degree of roasting to which the beans have been subjected (*vide* Chapters XI. and XXV.). Clayton † found from 0.83 per cent. to 2.69 per cent. of theobromine and from 0.02 per cent. to 0.66 per cent. of caffeine in commercial cocoa powders. Girard gives from 1.04 per cent. to 2.24 per cent. of theobromine.

\* Florence Yaple, *Chem. Zeit.*, 1895, xvi., 579.

† E. G. Clayton, *Chem. News*, 1902, lxxxvi. [2227], 51.

The estimation of theobromine in cocoa powders is of some importance, as the exhilarating and stimulating properties of cocoa are largely due to this alkaloid. The work of Prochnow \* has shown a higher percentage of xanthine bases in roasted than in unroasted cacao, thus—unroasted kernels 1·460 to 1·812 per cent. ; roasted kernels, 1·536 to 1·880 per cent. ; roasted husks, 0·578 to 1·380 per cent. †

STARCH (2·0 per cent. to 11·0 per cent. of cocoa powder).

The quantity of natural starch, found in cacao, is very variable. Its estimation, therefore, is of little value either as a check upon the amount of cacao present or upon the addition of foreign starches, unless the latter are present in large proportions and have been previously detected under the microscope.

Many observations of starch-content of cocoa powders have been made, but they are of little or no importance to the technical chemist or manufacturer.

The addition of foreign starches can readily be detected under the microscope, when the form of the granules of starches, likely to be used commercially as adulterants, can be recognised.

FIBRE (2·5 per cent. to 6·5 per cent. of cocoa powder).

It has previously been pointed out that the estimation of fibre is an indication to the extent of adulteration with, or inclusion of, cacao husk.

The inclusion of cacao husk, whether intentional or through carelessness, is a matter of great importance, as many convictions have followed the sale of "cocoa powder" consisting in part, or in the main, of cacao husk. Some such cases have already been referred to, and others, again, are considered in Chapter XXX.

\* A. Prochnow, *Arch. Pharm.*, 1909, cxxviii., 698.

† *Vide* also Chapter XI., p. 140.

Probably the most complete investigation into the detection of added husk has been made by Adan,\* who not only has estimated the cellulose- and pentosan-content of different varieties of cacao beans, but has carried out analyses of the cocoa powders, prepared from them. The direct connection between the estimation of pentosans and the amount of husk, present in any cacao, has been given in Chapter XXV., and in Table LXXV. will be found Adan's results for the nibs and husk of various cacaos.

TABLE LXXV.—*Cellulose- and Pentosan-Content of Cacao Nibs of known Origin.*

	Moisture.	Fat.	Ash.	Cellulose.	Pentosans.	
					Initial substance.	Powder with 30 per cent. of fat.
Arriba . . .	1.30	53.78	3.21	2.67	1.73	3.26
Port au Prince . .	0.83	55.60	3.56	3.14	1.58	3.73
San Thomé . . .	0.79	51.23	3.34	3.01	1.55	3.46
Caracas . . .	0.71	51.72	3.47	2.89	1.54	3.28
Bahia . . .	0.69	54.77	3.29	3.09	1.71	3.43
Soconusco . . .	1.01	55.98	3.13	2.91	1.57	3.23
Average . . .	0.88	53.86	3.33	2.95	1.61	3.40

Hehner and Skertchley † have estimated the pentosan- and crude fibre-content of several cocoa powders and cocoa shell powders. The following results, obtained by these two authors, are of interest :—

TABLE LXXVI.—*Pentosan and Crude Fibre in Cocoa Powders and Cocoa Shell Powders.*

	Pentosan.	Crude fibre.
Van Houten's cocoa . . .	2.00	6.00
Cadbury's cocoa . . .	1.82	7.15
Cocoa shell powder, I. . .	8.98	—
Cocoa shell powder, II. . .	8.03	11.95

\* R. Adan, *Internat. Cong. App. Chem.*, 1909, viii. C, 204.

† Hehner and Skertchley, *The Analyst*, 1899, xxiv., 178.

ALBUMEN (10 per cent. to 17 per cent. of cocoa powder).

The total nitrogen, found in cocoa powders, amounts, as a rule, to 3 per cent., or to an equivalent of, approximately, 19 per cent. of proteid matter, if all the nitrogen present is attributed to this component.

In reality, Dekker \* has shown that about 90 per cent. of the total nitrogen is due to proteid matter, the remaining 10 per cent. resulting from theobromine.

OXALIC ACID (0.4 per cent. to 0.65 per cent. of cocoa powder).

Girard, in his paper, frequently referred to, delivered at the Seventh International Congress of Applied Chemistry, has shown that the oxalic acid is largely present in cacao as alkaline oxalates, and his figures, which are the most reliable up to date, tend to show that the oxalic acid is not destroyed during roasting, or removed during expression of fat.

#### SUCROSE, CACAO-RED. ETC.

The remaining components of cocoa powders are of little importance to the technical chemist. Sucrose is found only in minute quantities, and only traces of reducing matters have been notified by the most reliable observers. This is of interest when chocolate and chocolate powders, to which cane-sugar has been added during preparation, are examined.

The colour of cocoa depends upon the cacao-red present, but the quantities, found in cocoa powders, are strictly comparable with the results given in Chapter XXVI. for the nibs, after allowance has been made for the removal of some 30 per cent. of the fat. The presence of cacao-red in nibs has provided a recent method for the detection of added husk to cocoa powders.†

\* S. Dekker, *Chem. Centr.*, 1902, ii., 1217.

† *Vide* Chapter XXXII.

### Plain Chocolate and Chocolate Powders.

The only differences that will be observed, on analysis, between chocolate, or chocolate powders, and cocoa powders are :—

(1) A large proportion of sugar, and some additional cacao butter, in chocolate.

(2) The presence of sugar and, probably, foreign starches, in chocolate powders.

The remaining components of cacao will be found in correspondingly decreased proportions.

FAT (22 per cent. to 28 per cent. of hard chocolate ; 28 per cent. to 38 per cent. of "chocolat fondant" ; 18 per cent. to 26 per cent. of chocolate powders).

The amount of fat in hard, plain chocolates seldom exceeds 28 per cent. It has previously been pointed out that the fatty chocolates ("chocolats fondants") are much in vogue, at the present time, and consist of ordinary cacao mass with sugar and a larger quantity of cacao butter than is to be found in the hard varieties. "Chocolats fondants" are, moreover, refined to a greater degree than the older-fashioned hard chocolates.

The chemist should ascertain the nature of the fat extracted from the sample of chocolate, as there are many cacao butter substitutes on the market.\*

In Chapter XVIII., Table XLI., will be found analyses by Booth of some adulterated chocolates. The same author gives the following mean analysis (of twenty-four samples) of pure, plain chocolates :—

Fat, 27·4 per cent. ; sugar, 53·4 per cent. ; nitrogen, 0·93 per cent. ; mineral matter, 1·32 per cent. ; cold-water extract, 56·8 per cent.

\* *Vide* Chapter XXIX.

SUGAR (45 per cent. to 55 per cent. of plain chocolates ; 50 per cent. to 60 per cent. of chocolate powders).

The amount of sugar, added, is, of course, optional to the manufacturer, though a quantity above 50 per cent. is difficult to work economically, as the chocolate is rendered less mobile and less easily moulded. A further addition of cacao butter, which is a costly ingredient, would have to be made, if the chocolate were not sufficiently fluid.

Sugar is (or was, and again will be) the cheapening agent, added in making chocolate, and it is, therefore, to the advantage of the manufacturer to work in as much as possible.

In Switzerland, a limit of 80 per cent. to 85 per cent. of fat and sugar together, the amount of sugar varying from 40 per cent. to 70 per cent. of the total chocolate, has been adopted as a standard.

Booth's recommendation that a legal standard for chocolate be adopted in England meets with our entire approval, though his suggested limit for sugar, namely, 65 per cent., seems to us too high.

Chocolate powders, used for drinking purposes, may contain larger quantities of sugar than plain eating-chocolates, as the preparation is usually in a granular or powdery form. Booth suggests a limit of 75 per cent. of sugar for chocolate powders. This figure we also consider to be too high, seeing that starch is nearly always added, as well, in the preparation of chocolate powders for drinking purposes.

#### STARCH.

The addition of starch to chocolate is not often met with, except in the cheapest preparations. As there is no legal standard for "chocolate" and its preparations, in this country, there is nothing to prevent the use of starch, though chocolates, prepared with it, can readily be detected on the tongue and are far from palatable. Starch may, however, be occasionally found in covering-chocolates.

At the present time, a mixture of cocoa with sugar and starch cannot be sold as pure "cocoa," but only as "chocolate powder," with a definite declaration that the article, sold, is a mixture of cocoa and other ingredients.

There are several highly nutritious cocoa preparations on the market, such as Epps's cocoa, prepared with selected arrowroot, sugar and cocoa. There is no objection to such preparations as these, provided that the public is fully aware what it is purchasing.

#### COLD-WATER EXTRACT.

The cold-water extract, obtained from chocolate or chocolate powder, depends, almost entirely, upon the amount of sugar present.

This value is remarkably constant in the case of cacao nibs (approximately 24 per cent. of fat-free cacao), and a higher figure indicates the probable addition of sugar. The actual percentage of sugar may be estimated by polarimetric methods, the cold-water extract of pure cacao having a negligible power of rotating polarised light.

#### Milk Chocolates.

In Chapter XX., will be found full analyses of milk chocolates by several chemists.

The average composition of milk chocolate is as follows :—

TABLE LXXVII.—*Composition of Milk Chocolates.*

	Booth.		Lara.	
	English.	Continental.	I.	II.
Total fat . . . .	31.80	30.80	31.47	32.33
Milk fat . . . .	5.50	8.10	4.97	5.93
Cacao butter . . . .	26.30	22.70	26.50	26.40
Milk sugar . . . .	8.04	8.26	7.32	8.70
Cane or beet sugar . . . .	43.20	42.60	27.51	35.93
Foreign starch . . . .	Nil.	Nil.	Nil.	Nil.
Cacao shell . . . .	Nil.	Nil.	Nil.	Nil.
Nitrogen . . . .	1.18	1.24	—	—

Laxa gives total proteid (I.) 9.66 per cent.; (II.) 8.13 per cent. The figures for milk fat and cacao butter are obtained by calculation (*vide* Chapter XX.).

FAT (total: 30 per cent. to 35 per cent. of milk chocolate).

It will be seen that the fat is capable of division into milk fat and cacao butter, the total fat being somewhat in excess of that from ordinary plain, hard eating-chocolates, and about equal to that obtained by extracting "chocolats fondants."

The chemical and physical constants of the fat, extracted from milk chocolate, will, of course, vary according to the ratio of milk fat to cacao fat.

MILK SUGAR (7.5 per cent. to 8.5 per cent. of good milk chocolate).

It must be understood that the value for milk sugar will vary in the same way as the milk fat- and nitrogen-content of the chocolate, according to the amount of milk, or milk solids, added.

The copper-reducing power of plain chocolate is small, as the sugar, added in the ordinary way, will be cane- or beet-sugar, which, if pure, will have no reducing power, whilst that of cacao nibs does not amount to more than 5 parts *per* 100 of dry and fat-free cacao.

Milk sugar, on the other hand, has a strong copper-reducing power, and its presence can be inferred by the action of the cold-water extract upon a copper solution.

#### NITROGEN AND ALBUMINOIDS.

The percentage of nitrogen and of albuminoids, or proteid matter, will be greatly increased by the addition of milk solids to chocolate, the former increasing from 0.9 per cent. to 1.2 per cent. and the latter from about 5.5 per cent. to about 8.5 per cent. in chocolates containing a good proportion of milk solids.



The estimation of nitrogen is, therefore, a useful added factor in determining the amount of milk solids present in a milk chocolate. The possibility of adding proteids from pea-nuts and soya has already been mentioned (p. 297).

### Milk Powders.

Some remarks on milk powders are not out of place here, as the rapid progress, made in the desiccation of milk, calls for the attention of the chemist and bacteriologist that would move with the times. The publications of the Local Government Board (Food Reports, No. 24, 1918, I., II. and III.) are extremely interesting, and this brief notice should be supplemented by these reports, by those further interested in the subject.

The methods of drying milk are confined to the "drum" and "spray" processes, of which there are numberless modifications. The most important of these are the processes of Campbell (1901), Ekenberg (1902 and 1903), Benenot and De Neven (1904), Just-Hatmaker (1903 and 1906), Kunick (1906 and 1907), "Trufood" (Merrell-Soule, patent 1906), "Glaxo," and Mignot-Plumey (1911).

The product of the "drum" process is a coarser material than that obtained with the "spray," though rancidity (if a full-cream milk has been prepared) occurs in both on exposure to the air, but faster in powders from the latter than in those from the former process. It has been stated that both sugar and cacao butter tend to prevent rancidity of the butter fat, so that rancidity does not develop very rapidly in milk chocolates in which both these ingredients are present.

Sir James Dobbie's report on the purity of commercial milk powders is interesting and states that all the samples, examined, contained proteids and lactose proportionately to those found in fresh milk. Some of the samples were stated to have been prepared with added lactose, so that

the proportion, added, cannot have been very high. Cane sugar was found in 5 out of 26 samples of full-cream milk powders, but only in such small amounts as 0.3 to 2.9 per cent. In skimmed or separated milk powders, 16 out of 57 cases contained added cane sugar, in amounts from 0.2 to 3.8 per cent. Bicarbonate of soda was occasionally found, in one case 7 grains to a pint of the original milk, but this had been added either to neutralise the original acidity of the milk or to render the powder more soluble in water. Added lime and phosphates were found in some samples. Boric acid was found in two samples, and salicylic acid in five. No added starch was detected, but traces of copper, occasionally, and, in one case, a minute trace of tin.

Dr. Coutts' conclusions on the "Bacteriology of Dried Milk" are important:—

"(1) It is clear that the processes used in drying milk largely reduce the number of bacteria present, but do not give an absolutely sterile product. (2) It is also clear that a considerable proportion of the bacteria found in dried milk, as it occurs in commerce, have been re-introduced in the concluding stages of the processes, frequently during the powdering and packing of the dry product. (3) With the object, therefore, of obtaining a product as free as possible from bacteria, it is important that special precautions should be taken to avoid re-contamination of the milk, and to this end the most scrupulous cleanliness should be observed in the factories in which dried milk is made. (4) Further, it is desirable that the milk should be drawn from the cow with due precautions to avoid access of dirt, and that it should be dried as soon as possible after milking to avoid the multiplication of germs in the liquid milk."

## CHAPTER XXVIII .

### MICROSCOPICAL EXAMINATION OF CACAO, COCOA AND CHOCOLATE

#### **Cacao Beans.**

Through the courtesy of Mr. A. S. Bryden, of Barbadoes, the author has been enabled to examine seeds of cacao, fresh from the pod. The samples of cacao pod, which were undoubtedly of Forastero origin, arrived in an air-tight tin in remarkably good condition. The contents of the pigment-cells, which contained a certain amount of violet colouring matter on arrival, had probably undergone an oxidation process during transit, as the beans, on cutting, showed a deeper violet coloration than would have been expected if cut perfectly fresh.

Thin sections of the bean were examined in drops of vegetable oil, as a distinct cloudiness was noticed on placing the sections in water, due probably to the fat which the beans contained.

Starting from the outside covering of the beans which were covered with a thick white slime of simple elongated cellular structure, the first and outer section of the shell of the bean was reached. The first epidermal layer consisted of a moderately thick skin containing polygonal cells, and, lying directly beneath this, were noticed the "exceedingly delicate transversely elongated cells," previously described by Ewell. The remainder of the shell consisted of closely compact bundles of spiral fibres and a series of tissues, bearing thick-walled cells, which seemed to contain a large

proportion of water. Within the shell was an exceedingly fine and transparent film or membrane, covering the whole of the kernel and falling into the spaces between the pieces, or nibs, of which the kernel is constructed.

Upon this transparent film, which is made up of a single layer of simple cells of irregular shape, the "Mitscherlich"



Photo, W. P. Puddison,

Brighton.

FIG. 30.—"Mitscherlich" Bodies between the Lobes of the Kernel.  $\times 300$  diam.  
(See p. 435.)

bodies grow (Figs. 30 and 31). These bodies are "chrysalis-shaped" and were supposed by Hassall to be of fungoid origin.

Hassall describes the thin membrane and the appearance of the "Mitscherlich" bodies in the following words: "Situated in the interspaces of the lobes is a fourth structure, attached externally to the second membrane, the cells

forming which pass down upon it for a short distance; although clear and transparent it exhibits a fibrous structure, and on its surface a considerable number of small crystals are always to be seen as well as many elongated bodies, rounded at their extremity and divided into several com-



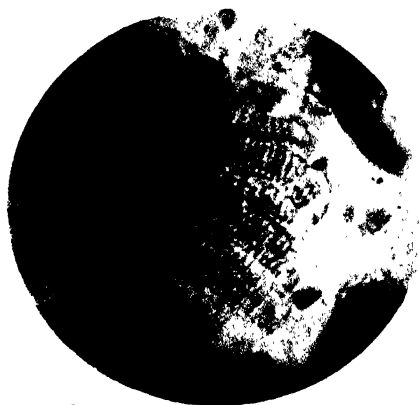
Photo, W. P. Paddison,

Brighton.

FIG. 31.— "Mitscherlich" Bodies, enlarged.  $\times 1,000$  diam. (See p. 435.)

partments or cells, and which do not appear to be attached to the membrane on which they lie. From their curious appearance and the absence of connection with any of the other structures of the cocoa seed the observer is led to suspect that they are extraneous and probably fungoid growths. We have detected them in every sample of cocoa seed submitted to examination."

Had Hassall seen these bodies in fresh cacao beans, he would have observed that they are attached to the thin membrane by an extremely small joint. It is probable that, during roasting and further treatment, the joint is broken, but that the bodies adhere to the thin membrane, as in all cases examined, after roasting, the bodies were found to be lying flat upon the membrane. The "Mitscherlich" bodies are now considered to be epidermal hairs,



Photo, W. P. Paddison,

Trighton

FIG. 32.—Cacao Husk, showing Bundles of Spirals. 1,000 diam. (See p 438.)

and, though they are said to occur in every part of the seed, their origin is undoubtedly from the interlobe membrane.

In the fresh bean, the section of the cotyledon or kernel appears as closely packed polygonal cells, filled with starch, fat and albuminoid matters, whilst, here and there, the strongly violet-coloured pigment-cells show up prominently. The colour rapidly fades or dissipates, and it has been found impossible to preserve a specimen showing the pigment-cells with the colouring matter *in situ*.

In the raw commercial and roasted kernel, the only real difference appears to be the change in the colouring matter,

which permeates every cell, from violet to brown. The spiral bundles, previously referred to as appearing in the shell, are also to be found in the kernel and are thrown into greater prominence by the processes of fermenting, curing and roasting, due to their being rendered opaque by loss of water (Figs. 32 and 33).

The starch granules, occurring in cacao, are very small and



Photo, W. P. Paddison,

Brighton.

FIG. 33.—Crushed Cacao Nibs.  $\times 1,000$  diam. (See p. 438.)

are not likely to be confounded with any other variety which might be used for adulterating cacao preparations. They are estimated by Ewell to range from 0.003 mm. to 0.10 mm.

In preparing microscopic slides of cacao and its preparations, glycerine jelly will be found to be an excellent medium, if the fat is not previously removed, and the cloudiness, already mentioned as appearing on placing the fat-containing

body in water, has not been noticed. Crystals of theobromine may often be seen by using this medium, though none were observed in sections taken from the fresh bean.

When using Canada balsam as a medium, the benzol, that it contains, dissolves the fat, which, on slow setting of the medium, crystallises out as fine, long needles. The benzol does not seemingly affect the theobromine crystals. It will be found desirable to de-fat the cacao and cocoa preparations before making permanent slides in Canada balsam.

Mitscherlich has recommended the addition of petroleum ether as the means of differentiation between crystals of cacao fat and theobromine. There can, however, be no real confusion on this point, as the fat crystals are almost always the longer and more slender, and their appearance is not often observed, except on the thin, transparent membrane, unless solvents are used to induce crystallisation.

The value of microscopical examination of cacao and cocoa preparations lies largely in the detection of adulteration, and the close study of the structure of the bean, though of great interest, is of little or no importance to the analytical chemist.

#### **Cocoa Preparations.**

Pure cocoa powder should appear under the microscope to contain no other ingredients than that of the cacao kernel.

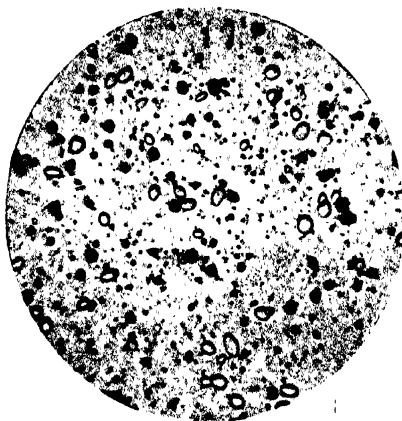
That is to say, pure cocoa powder will show cacao starch, the polygonal cells of the cacao kernel, traces of the transparent membrane covering the lobes of the kernel, and, possibly, the "Mitscherlich" bodies, already described, with a certain quantity of isolated spirals. Pure cocoa powder should not show foreign starch, the characteristics of which are too well known to the microscopist to need description, the sharp angular crystals of sugar, bundles of spirals, typical of those appearing in the husk, rounded transparent crystals of sand, or any of the cellular tissues characteristic of the husk of cacao.



Foreign starch, sugar, and cacao husk constitute the principal adulterants of cocoa powders, and their presence can readily be detected under the microscope (Fig. 34), the quantities being determined by chemical means.

The proportions of added materials can only be approximated by means of the microscope.

To chocolate powders and eating-chocolates the same remarks apply, though, in the former case, the addition of



*Photo, R. Whymper,*

*Dillon Hill*

FIG. 34.—Cocoa, adulterated with Arrowroot Starch. 100 diam. (See p. 440)

foreign starch and sugar is not prohibited, and, in the latter, sugar, only, should be permissible. Earthy matters can also be detected under the microscope; it is, however, improbable that such adulterants as red ochre, venetian red, etc., mentioned by Hassall, will be encountered at the present day.

Further than this, the purity of the added starch, sugar, or other ingredients can often be determined by examination under the microscope, and the fat, extracted from the preparation, can be examined independently, if crystallised from ether, many of the fats, with which cocoa and its

preparations are likely to be adulterated, having characteristic crystalline properties.

For further information regarding the microscopic structure of cacao, the valuable work of Moeler and Macé, "Les substances alimentaires étudiées au microscope," is recommended.

There are very few chocolates, appearing on the English and Continental markets, that are not examined microscopically in the author's laboratory. The following observations, selected from many made in 1914, may be of interest here :—

*Heldburg*.—Showed a remarkably fine and even appearance under the microscope.

The larger particles of sugar did not exceed  $\frac{1}{50}$  mm. The majority being  $\frac{1}{100}$  mm. or below. Possibly prepared from a syruped sugar.

The largest particles of cacao were seldom over  $\frac{1}{50}$  mm.

This chocolate was of a poor flavour, lacking character.

*Marquis*.—A very coarse and uneven chocolate.

The crystals of sugar were angular, rough and irregular, and were often  $\frac{5}{50}$  mm. The longest crystal was  $\frac{7}{50}$  mm.

The cacao was usually the size of the largest grains, *i.e.*, varying from  $\frac{5}{50}$ — $\frac{7}{50}$  mm.

This chocolate possessed a fine, full flavour and did not appear to have been closely refined.

*Kohler*.—Both the sugar and cacao particles were very small.

The sugar grains were never greater than  $\frac{1}{50}$  mm., mostly less than  $\frac{1}{100}$  mm.

The cacao grains, also, were very small, being usually equal to, or slightly greater than,  $\frac{1}{50}$  mm.

Flavour similar to "Velma."

*Cie. Coloniale*.—The sugar and cacao were both very coarse and very irregular.

The largest sugar grains equalled  $\frac{7}{50}$  mm. and were

usually about  $\frac{1}{10}$  mm., though the dimensions varied considerably.

The largest cacao grains were as large as  $\frac{9}{50}$  mm., but most of the cacao did not exceed  $\frac{4}{50}$  mm. and varied greatly.

This chocolate tasted of highly roasted cacao and was of a very fine full flavour.

*Meltis.*—Generally very regular, sugar not angular but small. The largest sugar particles were  $\frac{3}{50}$  mm.

The cacao grains showed a maximum of  $\frac{7}{50}$  mm. and were generally larger than the sugar grains. Fine flavour.

*Sandow.*—A fine, even chocolate under the microscope.

The sugar seldom exceeded, and was usually about,  $\frac{1}{50}$  mm.

Possibly prepared from syrupe sugar.

The cacao was about the same size as the sugar.

The chocolate tasted insipid.

*Lindt.*—The microscopic structure of this chocolate was somewhat irregular.

The largest sugar grains varied from  $\frac{1}{25}$  mm. to  $\frac{3}{50}$  mm., but were usually smaller. The grains were seldom angular.

The cacao was fairly fine, but many pieces were larger than the largest sugar grains by three or four times.

The sample was characteristic of the Lindt product, but was not of such full flavour as previous samples.

\* *Suchard Naps.*—The sugar was not very angular, but appeared smooth at the edges. The largest grains were  $\frac{3}{50}$  mm. to  $\frac{4}{50}$  mm.

The cacao grains were generally coarse and were equal in size to, or occasionally greater than, the largest sugar particles.

Of fine flavour.

*Golf.*—An exceptionally smooth and even chocolate.

The sugar was composed of minute rounded particles, which did not exceed  $\frac{1}{100}$  mm.

The largest cacao grains measured  $\frac{5}{16}$  mm., but were usually from  $\frac{1}{16}$  mm. to  $\frac{1}{32}$  mm.

This chocolate had a slight "Lindt" flavour.

*Velma Special.*—An irregular chocolate under the microscope.

The sugar grains were irregular and angular, measuring at greatest  $\frac{3}{16}$  mm.

The largest and usual size of cacao grains was  $\frac{4}{16}$  mm. and, very occasionally,  $\frac{1}{16}$  mm.

The particles of both sugar and cacao were generally smoother and finer than those of Suchard Naps.

Of a poor flavour, which had not been improved by moulding in thin, flat tablets.

*Cadbury's 1d. bar.*—Both cacao and sugar grains were large and uneven.

The sugar grains were angular and, occasionally, measured as much as  $\frac{4}{16}$  mm. Usually, the sugar grains measured between  $\frac{4}{16}$  mm. and  $\frac{1}{16}$  mm.

The cacao grains were large and uneven and measured, usually,  $\frac{1}{16}$  mm. to  $\frac{6}{16}$  mm., and, occasionally,  $\frac{5}{16}$  and  $\frac{1}{2}$  mm.

*Fry's 1d. bar.*—Abnormally large and uneven both in sugar and cacao grains.

The sugar was angular and often measured as much as  $\frac{12}{16}$  mm., and seldom was smaller than  $\frac{1}{16}$  mm.

The cacao grains were generally smaller than the sugar, but still large in size.

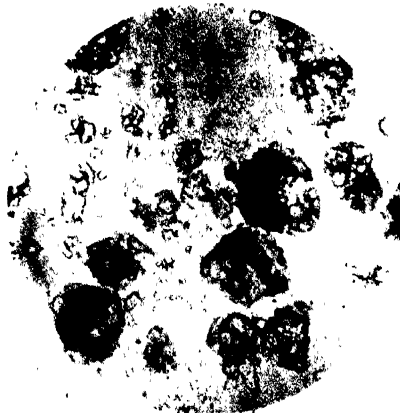
In carrying out researches on sugar, the degree to which chocolate is reduced by one, two or more refinings on steel and granite rollers, the effects of "conching," etc., a vast number of observations have been made under the microscope, and very many photo-micrographs have been taken by the present writer. Only four of these series are shown here, two illustrating the difference in appearance between ground crystal sugar (Fig. 35) and so-called "amorphous"

sugar, or transformed sugar (Fig. 36), one of a mixture of cacao mass, cacao butter and sugar, run down in a



*Photo, W. P. Paddison, Brighton.*

FIG. 35.—Ground Crystal Sugar.  $\times 70$  diam. (See p. 443.)



*Photo, W. P. Paddison, Brighton.*

FIG. 36.—Transformed Sugar.  $\times 70$  diam. (See p. 444.)

“melangeur” (Fig. 37), and one after this mixing has been refined once on a steel refiner (Fig. 38).

It will be observed, first of all, that crystal sugar, ground on mills or in a disintegrator, is composed of very fine particles of sharp-pointed pieces which, on each refining, will break down into yet other sharp-pointed fragments of a hard nature. It is these angular fragments that are felt as grit between the teeth, and it requires a number of refinings on steel rollers to reduce them to impalpable dimensions. The "amorphous" sugar particles, on the other hand, are



Photo, W. P. Paddison,

Brighton.

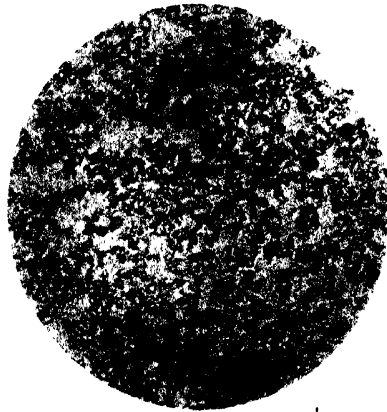
FIG. 37.—Chocolate Mass, made with Transformed Sugar, taken from the Mixer.  
 $\times 70$  diam. (See p. 444.)

much larger, individually, and the word *amorphous* is clearly a misnomer, as the particles are markedly crystalline; it is for this reason we prefer the word *transformed*. These transformed crystals are, however, of a very friable nature, being formed rapidly in the presence of a crystal inhibitor, and will break down, readily, even under pressure between the thumb-nails, into infinitesimally small particles.

This may be seen in Fig. 37, which shows a mixture containing this same sugar (illustrated in Fig. 36), cacao mass

and cacao butter, run for only twenty minutes in a "melangeur," during which the sugar has already been broken down into much smaller, irregularly shaped, but not sharp-pointed, particles. The cacao mass is even harder than the sugar, and the particles of the former are generally larger than those of the sugar.

One refining on the steel refiners reduces the cacao mass and the transformed sugar to an even size, of such small



Photo, W. P. Paddison,

Brighton.

FIG. 38.—(Chocolate Mass, as seen in Fig. 37, once Refined on Steel Refiners.  
× 70 diam. (See p. 444.)

dimensions that no grittiness can be felt between the teeth. One other refining is often necessary because, even on the best refiners, the rollers wear somewhat in use, and a certain amount of unground chocolate is apt to find its way through the worn parts of the cylinders. The result of one refining can be clearly seen in the last photograph (Fig. 38), which shows the same chocolate, illustrated in Fig. 37, after once passing through a 5-roll steel refiner.

"Amorphous" or shapeless sugar (that is, not apparently of definite crystalline formation) can be prepared by the

addition of a small quantity of lactose to the cane sugar in the process of transformation, outlined in Chapter XVI., and photo-micrographs of sugar, prepared in this way, are most instructive. Milk chocolate, manufactured by condensing milk with cane sugar, subsequently added to the cacao mass, owes much of its smoothness to this cause, and, though milk chocolate may have to be refined more than once, it is seldom due to the sugar particles, if the milk and sugar have been condensed down together.



## CHAPTER XXIX

### B.

#### METHODS OF ANALYSIS: FAT — DETERMINATION OF PHYSICAL AND CHEMICAL CONSTANTS OF CACAO BUTTER AND ITS SUBSTITUTES

THE analysis of cacao beans consists, essentially, in determining the amount of fat, moisture, ash, fibre, nitrogen and proteid matter that the nibs contain, the amount of husk obtained from the beans, and, if an assay of husk is required, the amounts of fat, moisture, ash, nitrogen and proteid matter that the shell contains.

An analysis of cocoa powder will require estimation of fat, moisture, ash, alkalinity of ash, fibre, nitrogen and proteid matter, cold-water extract and pentosans.

A preliminary microscopical examination will reveal husk, if much is present, and added starch and sugar, when, should either be present, a further estimation of these substances will be required.

A chocolate powder may require to be analysed for both added starch and sugar, besides the other components given under cocoa powders.

The analysis of a plain eating-chocolate will necessitate determination of fat (cacao butter), moisture, ash, alkalinity of ash, nitrogen and proteid matter, cold water extract, pentosans and sugar.

Fancy chocolates may require further determinations, such as lactose or milk sugar, milk fat and casein in milk chocolates, vegetable oils in nut chocolates, etc.

All chocolates may contain added starch and cacao butter substitutes, so that determination of the nature and quantity of both adulterants may be required.

Besides these estimations, those of theobromine, caffeine, cacao-red, etc., will be necessary, if a close analysis is to be made.

The methods of analysis of the essential components of cacao and all cocoa preparations will be described first.

In most cases, more than one method will be given, that first described being recommended.

Reference must be made at the outset to the generalisations of Bordas.\* This writer has stated that the various kinds of pure cacao pastes, used in the manufacture of chocolate, are sensibly the same, even though the cacao beans are derived from different sources. He then has proceeded to formulate his ideas on the composition of cacao and cocoa products in the following manner:—

(1) Percentage of non-fatty matter insoluble in water  $\times 1.30$  = percentage of dry, fat-free cacao.

(2) Percentage of non-fatty matter insoluble in water  $\times 2.95$  = percentage of dry, entire cacao.

(3) The quantity of butter, removed *per* 100 parts of the pure cacao, is found by the formula  $X = \frac{100(2.95I - A)}{2.95I}$ ,

where  $I$  = percentage of water-insoluble, non-fatty matter in the sample under investigation:  $A = 100$ , less the percentage of moisture in the sample.

(4) Cacao contains 2.9 to 3.8 per cent. of ash, the chief constituent of which is potash.

(5) Ordinary cacao contains, on the average, 2.54 parts of potash ( $K_2O$ ) *per* 100 parts of dry, fat-free cacao: those samples treated with alkalis, from 4.82 to 6.41 parts *per* 100 parts of dry, fat-free cacao.

(6) Cacao butter should have a saponification value 187—193, an iodine value 33—39, an oleo-refractometer deviation 18—19, and a Zeiss butyro-refractometer deviation 40—45.

\* F. Bordas, *Proc. Seventh Internat. Cong. App. Chem.*, 1909, viii. C, 187.  
w.c.

(7) Using invertase for hydrolysis, 0.65 per cent. of sucrose has been found in a mixture of pure cacao pastes, the use of acid for hydrolysis giving falsely high figures of 3—4 per cent. for sucrose, owing to the decomposition of glucosides, starch and inulin.

(8) Starch up to 28.5 parts *per* 100 parts of insoluble, fat-free matter were found by hydrolysing the material with acid or malt extract, after the fat and sugar had been removed, and after a treatment with 50 per cent. alcohol.

(9) By treating the residue on the filter after the starch estimation, first, with warm 0.1 per cent. solution of sodium hydroxide, then with water and alcohol, drying and weighing, the cellulose residue amounts to 18.8 to 16.6 parts *per* 100 parts of insoluble, fat-free matter.

It is clear that this method of expressing results can only be a generalisation and that more detailed information is required for a complete analysis.

#### Fat.

The separation of cacao butter from cacao and cocoa preparations has been the subject of considerable research.

The net result, obtained, is that the quickest and most complete solvent is petroleum ether, which removes the cacao fat without any theobromine. Other solvents of cacao butter, such as carbon bisulphide, ether and chloroform, remove a portion of the alkaloid also. Low fat results are invariably obtained if the cacao or cocoa preparation is not reduced to an extremely fine powder, to which end the sample may be ground with previously extracted, dry, quartz sand, or the precautions, recommended by Welmans,\* may be employed. In this method, he suggests that, in the cases of chocolate and cacao mass and other fatty preparations, the sample be rubbed down in a mortar at 50° C., until the coarser particles are no longer visible.

\* P. Welmans, *Zeitsch. öffent. Chem.*, vi., 304.

The fluid mass is allowed to solidify in tin moulds and cooled; when cold, the mass is rubbed on a grater, and the whole operation repeated. By this method, a fine, homogeneous sample is obtained.

#### *Method I.*

Five grammes to 10 grammes of the finely divided sample are placed in a fat-free filter cone and extracted with petroleum ether in a Soxhlet extractor. After the ether has passed over from 12 to 15 times, the flask, which had previously been weighed and which now contains the ether and fat, is subjected to gentle heat, and the ether is distilled off. The flask, containing the fat, is then placed in the steam oven, until no further loss in weight is observed, and the percentage of fat calculated.

It should be noted that Keller\* has found that a sixteen-hours' treatment in a Soxhlet is sufficient to remove all the fat from cocoa powders, without the addition of sand which is often recommended to facilitate extraction. He points out, also, that a small, though almost negligible, amount of theobromine is also removed by the ether.

#### *Method II.*

P. Welmans recommends the following procedure:—

Five grammes of the finely divided powder are agitated in a separator with 100 ccs. of ether-saturated water, till coherent particles are no longer observable; 100 ccs. of water, saturated with ether, are then added, and the mixture briskly agitated, till an emulsion is formed.

The mixture is allowed to rest and separate into two layers. Twenty-five ccs. to 50 ccs. of the clear ethereal solution on the top are removed by means of a pipette, the ether distilled off, and the residue weighed.

Prochnow† adopted a modified Welmans process, but

\* Keller, *Apoth. Zeit.*, 1916, *xxi.*, 330.

† A. Prochnow, *Arch. Pharm.*, 1910, *ccxlviii.*, 81.

the finely divided\* powder was extracted with ether in a Soxhlet apparatus for 18 hours. At the end of that time, the extracted powder was removed from the cartridge, rubbed down in a mortar and again extracted for three hours. In several cases, an additional quantity of fat, amounting, occasionally, to 1.5 per cent., was extracted by the second treatment. The percentage of fat, found, varied in raw beans from 50.80 to 53.98 per cent., and in the roasted beans from 50.12 to 53.70. Prochnow, in criticising the rapid methods of Tschaplowitz\* and of Kirschner† for estimation of fat in cocoa preparations, has stated that such processes are only applicable for approximate analyses.

### Method III.

A. Kreuz‡ makes use of chloral alcoholate as a solvent in the following method :—

Two grammes to 3 grammes of the sample and 3 grammes of chloral alcoholate are placed in a flask and heated to a homogeneous mass. To this mixture, 15 ccs. of ether are added, and the contents of the flask well shaken ; a further 35 ccs. of ether are added, and the whole filtered into a weighed flask. The residue in the filter is washed twice with pure ether. The flask and ethereal solution of fat is dried at 105° to 110° C., at which temperature the chloral alcoholate is completely expelled.

In a subsequent communication, he recommends the removal of the chloral alcoholate by heating under reduced pressure at a temperature of about 75° C. The residue, so obtained, is treated with chloroform to re-dissolve the fat, the solution filtered, to separate the theobromine and cacao-red, the filtrate evaporated, and the fat weighed.

\* Tschaplowitz, *Jour. Chem. Soc., Abstr.*, 1906, ii., 404.

† Kirschner, *ibid.*, ii., 502.

‡ A. Kreuz, *Zeitsch. Nahr. Genussm.*, 1908, xv., 680, and xvi., 584.

## Method IV.

Davies and McLellan\*, and Steinmann†, recommend the use of rectified petroleum ether, boiling at 60° C., as the solvent, the last mentioned author pointing out that the errors, caused by making use of Kreuz's method (III.), are considerable, especially in the case of chocolates. The Soxhlet method is recommended by these workers.

The use of ethyl alcohol, for obtaining a finely divided sample for subsequent extraction, is recommended by many observers.‡

There are various modifications of the ether-extraction method, devised, often, with a view to accelerate the estimation. One such method, proposed by Hughes§ is based on Bordas and Touplain's method for the rapid valuation of milk.

The materials, to be analysed, are, firstly, ground as finely as possible and passed through a 30-mesh sieve. Two grammes of the sample are then taken, and 30 ccs. of 50 per cent. alcohol added, the whole mixed thoroughly and whirled on a Leffman-Beam centrifugal machine in glass cylinders holding about 40 ccs. The clear liquid is decanted off and rejected, and a second application of about 30 ccs. of 50 per cent. alcohol made, the whole whirled, and the clear liquid again rejected. To the residue, after alcoholic extraction, a mixture of equal volumes of ordinary ether and petroleum ether is added, stirred, and well mixed for about 15 minutes and whirled, the clear liquid being decanted into a tared flask. A second extraction with the ether mixture is made, and this is usually found to be sufficient to effect complete extraction of the fat which, the author claims, is free from impurity.

\* S. H. Davies and B. G. McLellan, *Jour. Soc. Chem. Ind.*, 1904, 480.

† A. Steinmann, *Chem. Zeit.*, 1905, xxix., 1074.

‡ Tschaplowitz, *Zeitsch. anal. Chem.*, 1906, xlv., 231; A. Kirchner, *Zeitsch. Nahr. Genussm.*, 1906, xi., 450, etc.

§ E. B. Hughes, *Chem. News*, August, 1919, cxix., 104.

Hughes gives results, obtained by his method compared with those by Soxhlet extraction, as follow :—

	From Whole-fat cocoa. Fat per cent.	From Commercial cocoa. Fat per cent.	From Chocolate. Fat per cent.
Centrifugal method	44.31	21.15	31.55
Soxhlet method	44.37	20.85	31.50

A centrifugal method for separation of the solvent and solids, in which ether is used, and in which the collected solution is evaporated and dried, is official in France.\*

Another method, which calls for some attention and further experiment, is that devised by Richter.† The Zeiss Refractometer is used, in this method, on an ether infusion of the material to be examined, extracted by an ether and alcohol trisodium phosphate solution. Equal volumes of alcohol and ether are mixed together, and the trisodium phosphate is prepared by adding 65 grammes of the salt to a litre of water. Preliminary experiments are necessary to ascertain how much of the ether-alcohol mixture must be added to 100 ccs. of the phosphate solution, so that 5 ccs. of ether will separate after mixing. The proportions of ether and alcohol in the mixture are so adjusted that 25 ccs. of the mixture must be added to yield 5 ccs. of ethereal layer.

Ten grammes of the powdered cocoa preparation are taken and mixed with the found quantity of ether-alcohol mixture; 100 ccs. of the phosphate solution are then added. The whole is shaken for some ten minutes and allowed to settle. The extracts of chocolates, and of coarsely powdered cocoas, should be slightly warmed and, subsequently, cooled to 17.5° C., at which temperature all measurements are made. The clear ethereal solution is

\* *Ann. des Falsif.*, 1911, iv., 417.

† O. Richter, *Zeitsch. Nahr. Genussm.*, 1912, xxiv., 312.

examined in the refractometer and the percentage of fat ( $F$ ), present, calculated from the formula—

$$F = \left( \frac{n - n_1}{n - n_2} - 1 \right) \frac{100 XY}{Z},$$

where  $n$  = refractive index of cacao butter = 1.4653,

$n_1$  = refractive index of solvent,

$n_2$  = refractive index observed for the fat solution,

$X$  = the excess of volume of the ether (5 ccs.),

$Y$  = S. G. of the fat,

$Z$  = weight of the substance in grammes (10 grammes).

TABLE LXXVIII.—*Co-operative Results on Ether-Extract by the short-method, testing effect of Temperature and Necessity of Filtration (see p. 456).*

Analyst.	Filt- tered.	Not filt- tered.	By continuous extraction		Tem- pera- ture.	Remarks.
			Petro- leum ether.	Ether.		
	Per cent.	Per cent.	Per cent.	Per cent.	° C.	
C. J. Lott	23.09	22.80	—	—	—	
M. C. Albrecht	—	23.01	—	—	29	
L. C. Mitchell	23.13 23.05	22.81 22.58	—	23.35 23.26	—	
N. Hendrickson	23.56 23.53 (24.46 23.43 23.43)	—	—	—	27 20.5 20.5	
H. S. Bailey	23.86 23.06 25.07 (24.63 23.85 23.20)	—	—	—	22 24 29 20	
	—	22.97	—	—	25	
	23.41	—	—	—	28	
B. S. Hiltner	—	23.06	—	22.98	—	Knorr apparatus; sample not ex-tracted, fat not per- fectly clear.
	—	—	—	22.87	—	Johnson extractor; four hours, fat not perfectly clear.
	—	—	—	22.64	—	Leach-Hiltner method
	23.49 24.39	22.64	—	—	18.5	
W. L. Dubois	—	—	—	—	29	
	—	—	22.35	—	—	Alundum thimble in Knorr apparatus. Ditto.



The work of the U. S. Department of Agriculture, (*Bur. of Chem. Bull.* 152) in 1911, provided some interesting figures in connection with rapid determination of fat in cocoa preparations. Two objections had been found to the method suggested in 1910 :—

- (1) Loss by evaporation of the solvent during filtration.
- (2) That higher values were obtained on hot days than on cold, owing to the evaporation of the solvent. The objections were considered by a referee, who provided a sample of cocoa for the estimation of fat by different collaborators in different laboratories. Table LXXVIII. shows the results, obtained.

In the discussion that followed, the referee stated that this method (see suggested A.O.A.C. rapid method, 1910) could only be recommended for approximate determinations and in cases when rapid analyses were desired. The substitution of petroleum ether for sulphuric ether was further recommended in view of the fact that theobromine and certain quantities of substances, other than cacao butter, were found to be extracted by sulphuric ether and not by petroleum ether, whilst the fat was equally soluble in both solvents. Results on five cocoas were shown, thus :—

*Comparison of Ether-extract Results, using Sulphuric  
and Petroleum Ether.*

Sulphuric ether Per cent.	Petroleum ether. Per cent.
23.07	22.74
29.39	28.86
28.12	27.52
24.99	24.40
23.15	22.56

In 1912, a further report (*U. S. Dept. Agric. Bull.*, 162, 1912) confirmed the desirability of using petroleum ether, and a provisional method was put forward.

A rapid method, proposed by Kreis,\* is to boil 1 gramme of cocoa for fifteen minutes in a graduated tube with 20 ccs. of 1.5 per cent. hydrochloric acid. The mixture is cooled to 30° C., shaken for five minutes with ether and centrifuged for fifteen minutes (at least 1,000 r.p.m.). An aliquot part, say 25 ccs., of the ethereal solution is transferred to a tared nickel basin, the ether evaporated, the residue of the fat heated for ten minutes in a drying oven and weighed. After the quantitative extraction of a small, carefully weighed sample of cacao or cocoa preparation, a larger quantity must be extracted for the qualitative tests to be performed upon the fat.

The high price of cacao butter has caused the frequent substitution of other vegetable fats, and, though not prohibited in this country, owing to the absence of legal standards, it is certainly desirable to ascertain the nature of the fat employed.

The substitutes of cacao butter that are most likely to be encountered are given, with their constants, in Table LXXX. There are, however, a number of qualitative tests, which can be rapidly performed, which afford a useful indication as to the purity of the cacao butter, before the more elaborate methods of determining its constants need be used. The most important of these is Björklund's ether test.

#### BJÖRKLUND'S ETHER TEST.

The ether test, which, in a modified form, constitutes the British Pharmacopœia test of purity, consists in shaking a quantity of the fat (3 grammes.) in a test-tube with twice its weight of ether at 18° C. Pure cacao butter produces a clear solution, whilst the presence of wax will at once cause turbidity, which will not clear, even on warming.

\* H. Kreis, *Chem. Zeit.*, 1916, xl., 832.

If a clear solution is obtained, the test-tube is immersed in water at 0° C., and the minutes which elapse, before turbidity occurs, noted.

Björklund made the following observations:—

	Turbidity at 0° C. after minutes.	Clear solution at degrees C.
Pure cacao butter . . . . .	10—15	19—20
Cacao butter + 15 per cent. beef tallow	8	22
Cacao butter + 10 per cent. beef tallow	7	25

It has been observed, also, that the form of the crystals in the chilled, ethereal solution is a useful indication of the addition of tallow, pure cacao butter crystallising in well-defined shapes at the bottom and sides of the tube, whilst a small percentage of tallow renders the solution cloudy and flocculent.

#### REFRACTIVE INDEX ON ZEISS BUTYRO-REFRACTOMETER.

The determination of the refractive index of a fat has long been known as a useful indication of the state of purity.

The instrument, usually employed, is the Zeiss butyro-refractometer, which, from its handiness and for general utility, is most to be recommended.

There are a few oils, however, such as tung oil and the resin oils, which fall without the scale (5° to 105° on the butyrometer), and, for these, the Abbé refractometer, or the oleo-refractometer of Amagat and Jean, may be used.

The Zeiss butyro-refractometer, which consists of an Abbé double prism, capable of being heated by a current of hot water, and a permanently attached telescope, the objective of which is adjusted by a micrometer screw, does not need further description, the method of working being one of greatest simplicity.

Some figures of value for the detection of adulteration of cacao butter, obtained by the author, are given:—

	Readings on Zeiss butyro- refractometer at 40° C.
Cacao butter . . . . .	46—47
Coconut stearin . . . . .	35—36
Beef tallow . . . . .	48.5—49.0
Palm butter (hard) . . . . .	47.0
Palm-kernel oil . . . . .	38—39
Earth-nut oil . . . . .	55—56
Butter fat . . . . .	42—45 (usually 44.5)
Hazel-nut oil . . . . .	54.2
Almond oil. . . . .	57.2
Fat from Cailler's milk chocolate . . . . .	46.0
Fat from Nestlé's milk chocolate . . . . .	46.0
Fat from Peter's milk chocolate . . . . .	46.2
Fat from "Nuttis" (Peck Frean's nut chocolate) . . . . .	50.0

It has been thought desirable to include, for consideration, only those physical and chemical constants of cacao butter which are of the greatest importance and value to the analyst, and which will enable him to detect the presence and extent of adulteration with foreign fats. For this purpose, the methods of estimation of the specific gravity, the melting-point, the melting-point of the fatty acids, the saponification value, the Reichert-Meissl value and the iodine value, only, will be discussed. If further information, concerning the estimation of other values for cacao butter, is required, Allen's "Commercial Organic Analysis," vol. ii., 1910, is to be recommended.

In the works of Alder Wright, Lewkowitzsch and others on oils, fats, waxes, etc., will also be found full descriptive details for the processes involved in estimating acid value,

Hehner value, acetyl value, bromine value, refractive index, etc., which may be required, from time to time, for special cases.

#### SPECIFIC GRAVITY.

Fat possesses a specific gravity largely depending upon its constitution. The determination of this value, therefore, is of some importance in detecting adulteration of cacao butter with any particular oil or fat.

In the case of an oil, the determination of specific gravity can be made by means of a hydrometer, specific gravity bottle, or Sprengel tube at any moderate temperature, compared against that of water at  $15.5^{\circ}\text{C}$ ., which, in England, is usually taken as the unit of comparison.

In the case of a fat or wax, solid at ordinary temperatures, such as cacao butter, the fat is melted, and the specific gravity is taken at some higher temperature, preferably the boiling point of water ( $100^{\circ}\text{C}$ .).

The Sprengel tube, which gives more accurate results than the hydrometer or specific gravity bottle, is constructed of thin glass tubing, having an internal diameter of about  $\frac{1}{2}$ -inch. The tube is usually bent into a U-shape, terminating at both ends in capillary tubing.

The weights of the Sprengel tube and of the volume of water it contains at  $15.5^{\circ}\text{C}$ . are first ascertained. The clean, dry tube is then filled with the molten fat, by immersing one end of one capillary tube in the liquid and applying suction to the other. The whole is then placed in a beaker of boiling water, in such a way that only the extreme tips of the capillaries are not covered. As the temperature of the molten fat rises, expansion occurs, and the oil drips from the fine orifices of the capillary. As soon as expansion has ceased, and the oil has attained the temperature of the boiling water, the Sprengel tube is removed, carefully dried, cooled and weighed. The weight of the contents divided by the weight of water which the tube originally contained at  $15.5^{\circ}\text{C}$ . will

give the specific gravity of the oil at 100°C. compared with water at 15.5°C.

Table LXXIX. shows the specific gravities of various fats, solid at the ordinary temperatures, as observed by Allen. Though these results were obtained by a different method (the plummet method \*), they could have been ascertained by the Sprengel tube method, described, with equal facility and greater accuracy.

TABLE LXXIX.—*Specific Gravities of Cacao Butter and some possible Adulterants.*

	Specific gravity of melted fats, etc. water at 15.5° C. (1,000)		Difference for 1° C.
Cacao butter . . . . .	892.1 at 50° C.	857.7 at 98° C.	0.717
Palm oil . . . . .	893.0 at 50° C.	858.6 at 98° C.	0.717
Japan wax . . . . .	901.8 at 60° C.	875.5 at 98° C.	0.692
Tallow . . . . .	895.0 at 50° C.	862.6 at 98° C.	0.678
Butter fat . . . . .	904.1 at 40° C.	867.7 at 99° C.	0.617
Coconut stearin . . . . .	895.9 at 60° C.	869.6 at 99° C.	0.674
Coconut oil . . . . .	911.5 at 40° C.	873.6 at 99° C.	0.642
Palm nut oil . . . . .	911.9 at 40° C.	873.1 at 99° C.	0.657
Paraffin wax . . . . .	780.5 at 60° C.	753.0 at 98° C.	0.724

The specific gravity of a fat may vary within narrow limits, according to the method of expression or extraction, the presence of fatty acids, the age of the fat, and other circumstances.

#### MELTING- AND SOLIDIFYING-POINTS.

The addition of extremely small quantities of oil will rapidly lower both the melting-point and the solidifying-point of cacao butter. The reverse is the case on addition of a fat or wax of higher melting-point.

The true melting-point of cacao butter lies between 30° and 34° C., when it fuses to a transparent yellow liquid, which congeals again at 20.5° C.

*Vide Allen's "Commercial Organic Analysis," 1910, ii., 48.*

## MELTING-POINT.

*Method I.*

The cacao fat is first melted, at about 33° C., and drawn up into a fine, thin capillary tube, made by drawing out a piece of ordinary quill tubing. The fat is made to solidify immediately in the tube, which is then placed away for two or three hours. The longer the fat is allowed to remain solidified up to, say, twenty-four hours, the more nearly will the true melting-point of the fat be obtained. The capillary tube, which is open at both ends, is attached to an accurate thermometer, at its bulb, by a thin piece of rubber band or tubing.

The thermometer and attached tube are then immersed in water, which is *very gradually* heated and constantly stirred, until fusion of the fat takes place. The mean of the temperatures at the point of fusion of the fat, observed over a number of experiments, may be taken as the melting-point.

The flame is then removed from beneath the receptacle containing the water, and the temperature at which the fat solidifies observed, as the water cools.

As a rule, the solidifying-point of a fat is a very much lower temperature than the melting-point—this is the case with cacao butter.

*Method II.*

The method of determining the melting-point of fats, adopted by the A.O.A.C., is somewhat more complicated, though extremely accurate.

The fat, melted and filtered, is made to fall from a dropping tube, from a height of 15 to 20 cms., on to a smooth piece of ice floating in distilled water. The discs of solid fat, so formed (1 to 1.5 cms. in diameter), weigh about 200 mgrms., and are removed by pressing the ice under the water, when they float to the surface and can be readily removed.

The apparatus, employed, consists of a boiling-tube,

35 cms. long and 10 cms. diameter, containing a smaller test-tube, 30 cms. long and 3.5 cms. diameter. A small-bulbed thermometer and fittings for blowing air into the outer tube complete the apparatus.

Some distilled water and 95 per cent. alcohol are first separately boiled, to remove any traces of air which they might contain in solution. The hot water is then poured into the inner tube, until the latter is half full, when the hot alcohol is admitted slowly down the side, until the tube is about three-quarters filled.

The object of these precautions is, firstly, to prevent the formation of bubbles of air on the disc of fat, which is subsequently placed in the inner tube, and, secondly, to avoid complete mixing of the alcohol and water. The inner tube is then placed in the boiling-tube containing ice-cold water, until the contents of the former are quite cold.

When cold, the disc of fat is placed in the inner tube containing the alcohol and water, into which the disc sinks, until the specific gravity of the mixture is the same as that of the fat.

The delicate thermometer, with a fine bulb, is let down into the inner tube so that the bulb is just above the disc. The temperature of the water in the boiling-tube is now gently raised, and the water kept constantly stirred by blowing in air, from a rubber blowing bulb, at the bottom of the boiling-tube.

When the temperature has been raised within a few degrees of the melting-point of the fat, the disc of fat will be seen to shrivel, and, at this point, the thermometer should be lowered so that the level of the bulb is the same as that of the disc, and a gentle stirring motion given, to cause a uniform temperature in the neighbourhood of the disc. As soon as the fat disc has assumed an almost spherical shape, the temperature of the solution, in which it is immersed, is taken at the level of the disc.



A second and third repetition of this operation will give very accurate readings for the melting-point of the fat.

#### SOLIDIFYING-POINT.

The determination of the solidifying-point of the fats is of less importance and may be made after the determination of the melting-point, described under Method I. It is desirable, however, to substitute a capillary tube, closed at one end, for that open at both ends, as used in this method.

The value of estimating the melting- and solidifying-points of the fatty acids obtained from the fats is considerable, and the variations, occurring in the figures of the two estimations, are less than when the natural fats are employed.

#### *Method I.*

A modification of Dalican's method, official in the United States, is extremely accurate and embodies the original method, suggested by Dalican, with the precaution that constant quantities of chemicals for the preparation of the fatty acids, standard dimensions of apparatus, etc., are employed, so that the solidifying-point of the fatty acids can be determined under strictly comparable conditions.

Twenty-five grammes of fat are saponified with 60 ccs. of 30 per cent. sodium hydroxide solution (36° Bè.) and 75 ccs. of 95 per cent. alcohol, or 120 ccs. of water, in a platinum dish, and the mass evaporated to dryness.

The dried soap is dissolved by 1,000 ccs. of boiling water, and the solution boiled, until all the alcohol has been expelled (usually about 40 minutes). The solution is then treated with 100 ccs. of 30 per cent. sulphuric acid (25° Bè.) and heated, until the fatty acids, set free, are quite clear.

The fatty acids are then thoroughly washed with fresh quantities of distilled water, separated in a separating funnel, filtered through a dry filter-paper and thoroughly dried for 20 to 30 minutes in a steam-heated oven at 100° C.

The dried fatty acids are allowed to cool to  $15^{\circ}$  or  $20^{\circ}$  C. above the suspected melting-point; they are then poured into a test-tube 100 mms. long and 25 mms. in diameter.

This test-tube is made to fit into an empty flask, 150 mms. long by 70 mms. in diameter, by means of a cork, and a thermometer, registering from  $10^{\circ}$  to  $60^{\circ}$  C., and graduated in tenths of a degree, is passed through a hole in a cork of the inner tube, until the bulb reaches the middle of the material. The  $10^{\circ}$  mark on the scale of the thermometer should be about 3 cms. above the bulb, and the entire length of the thermometer should not exceed 36 cms.

When the fats, or fatty acids, commence to solidify at the bottom of the tube or around the sides, the thermometer should be given a gentle stirring motion, to equalise the temperature of the material. At first, the temperature will be seen to fall slowly, and then to rise to a point, when the mercury will remain stationary for one to two minutes. The readings of the thermometer should be taken at short and equal intervals of time, and the highest temperature, recorded during the short rise following the fall of the mercury, gives the solidifying-point or "titer" of the material.

The experiment should be repeated 2 or 3 times, and the readings, obtained, should not vary by more than  $0.1^{\circ}$  C., if proper care has been exercised.

#### *Other Methods.*

There are several other well-recognised methods of determining the solidifying-point of fatty acids, etc.

Finkener's\* method, which had an official status in Germany for the differentiation of lard, tallow and candle fats, Wolfbauer's† method, Shukoff's‡ method, and others,

\* Finkener, *Chem. Zeit.*, 1896, xx., 132.

† Wolfbauer, "Mitt. techn. Gew. Mus. in Wien.," 1894, 57

‡ Shukoff, *Chem. Zeit.*, 1901, xxv., 1111.

can be found described in Allen's "Commercial Organic Analysis," 1910, vol. ii., 56—58. or in the original papers given in the footnotes.

It has been found that the method and duration of saponification have no material influence on the results obtained by any of the foregoing methods, the most important factors, in obtaining consistent results, being the complete expulsion of alcohol after saponification and the thorough drying of the fatty acids.

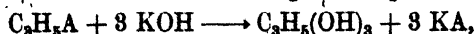
For the methods of determining other physical constants of cacao butter, such as action on polarised light, electrical conductivity, viscosity, heat of combination, etc., Allen's "Commercial Organic Analysis," vol. ii., Lewkowitsch's and Alder Wright's books on oils, fats, waxes, etc., should be consulted.

#### SAPONIFICATION VALUE OR KOETTSTORFER \* VALUE.

The saponification value of an oil or fat is expressed as the number of milligrammes of potassium hydroxide required for the complete saponification of 1 gramme of that oil or fat.

Oils and fats, of the nature which will come under the notice of the chocolate manufacturer and his chemist, are, with the exception of paraffin wax, broken up by the action of alcoholic alkali into the alkaline salt, or soap, of the fatty acids, existing in the fat, and glycerol or glycerin, which existed originally in the fat as the glyceride, or glycerol compound, of the fatty acids:

The saponification of fatty oils is a perfectly definite reaction, and not only can the proportion of fatty acid and glycerol, produced from any fat or glyceride, be determined, but the actual amount of alkali involved in the reaction can be ascertained. Thus, in the general equation—



\* Koettstorfer, *Zeitsch. anal. Chem.*, 1879, xviii., 199.

the fat or glyceride ( $C_3H_5A$ ), containing a fatty acid A., combines with potassium hydroxide (KOH) to give glycerol ( $C_3H_5[OH]_3$ ) and the potassium salt, or soap, of the fatty acid A, (KA).

In the above equation, the number of parts of fat saponified by 1 molecule of potassium hydroxide will be one-third of its molecular weight, but, in the case of an ester of a monatomic alcohol, the number will be identical to its molecular weight. This figure is, briefly, what is termed the "saponification equivalent" and represents the amount of oil or fat, in grammes, which would be saponified by 1,000 ccs. of a normal solution (N) of any alkali.

In determining the saponification value, however, the amount of fat is the constant, and the amount of alkali is the variable.

#### Method I.

From 1.2 to 2 grammes of the fat are weighed out and treated with an accurately measured 25 ccs. of approximately semi-normal  $\left(\frac{N}{2}\right)$  solution of potassium hydroxide in alcohol, in a flask bearing a long tube to act as an air-condenser.

The alcohol should be previously freed from water by keeping an excess of dry potassium carbonate in the bottle containing it.

The flask and its contents are heated on a water-bath, until solution of the fat has taken place, and the saponification is complete. This usually takes from 30 to 45 minutes.

In order to ensure greater accuracy in the subsequent titration, a test experiment should be conducted side by side with the fat undergoing saponification. The test experiment consists of measuring out accurately 25 ccs. of the same approximately  $\frac{N}{2}$  potassium hydroxide solution previously added to the fat, and heating it, in a flask

similarly constructed to that in which the fat is being saponified, for the same length of time.\*

One cubic centimetre of a 1 per cent. alcoholic solution of phenolphthalein is then added to each flask, and the liquids titrated with  $\frac{N}{2}$  hydrochloric acid.

The difference between the volumes of standard acid, used in the two cases, will give the number of cubic centimetres corresponding to the alkali neutralised in saponifying the oil, each cubic centimetre of  $\frac{N}{2}$  hydrochloric acid (18.25 grammes of hydrochloric acid in 1,000 ccs. of solution) representing 0.02805 grammes of potassium hydroxide (KOH). From these figures, the number of milligrammes of potassium hydroxide, required to saponify 1 gramme of the fat, can readily be obtained.

From the saponification value, so found, the saponification equivalent can be calculated by dividing the former into 561, and multiplying the result by 100.

#### *Method II.*

A method of cold saponification may, at times, be found useful.

In this method, 3 to 4 grammes of the fat, dissolved in 25 ccs. of re-distilled petroleum ether, are treated with 25 ccs. of normal (N) alcoholic potassium hydroxide solution, and the mixture, after thorough shaking, is kept in a flask for 24 hours. The excess of alkali is titrated with standard hydrochloric acid, as previously described. A blank experiment must be conducted side by side with the fat undergoing saponification, in order to check the strength of the alcoholic potassium hydroxide employed, which will vary from day to day.

#### REICHERT-MEISSEL VALUE.

The Reichert-Meissl value, which is of special importance

in butter analysis, is a modification by Meissl\* of Reichert's† method for the estimation of the number of cubic centimetres of decinormal  $\left(\frac{N}{10}\right)$  alkali solution required to neutralise the distillate obtained from an acidified solution of a fat, previously saponified, under definite conditions.

Reichert's original method need not be discussed here, but will be found, if required, in the paper mentioned in the footnote.

#### Method I.

Five grammes of the fat are carefully weighed out, and cautiously heated, with constant shaking, over a small flame, in a 300 cc. Erlenmeyer flask with 20 ccs. of glycerol (specific gravity, 1.26), and 2 ccs. of sodium hydroxide solution (100 grammes sodium hydroxide in 100 ccs. of water). The water is then evaporated off, an operation which usually takes 5 to 8 minutes, resulting in the formation of a clear liquid.

The flask and contents are warmed over a steam- or water-bath, until the fat is completely saponified. It is then allowed to cool to about 80° C., and treated with 90 ccs. of distilled water at the same temperature.

The solution is then acidified with 50 ccs. of dilute sulphuric acid (50 ccs. of strong acid in 1,000 ccs. of water), and the volatile fatty acids, set free, are distilled off.

Before distillation, care should be taken that the fatty acids are quite molten and that the globules in the emulsion have fused together. This may be brought about by heating the flask and contents on the water-bath, until the fatty acids appear as a transparent, oily layer on the surface of the water.

*Distillation.*—After the fatty acids have separated into a clear layer on the surface of the water, the flask is cooled

\* Meissl, *Dinglers Polyt.*, 1879, cccxxxiii., 229.

† Reichert, *Zeitsch. anal. Chem.*, 1879, xviii., 68.

to the temperature of the room, and a few pieces of pumice-stone added. The pumice-stone should be prepared by heating it to a white heat and throwing it into distilled water, in which it is kept immersed, until wanted.

The flask is now connected to a glass condenser and slowly heated over a naked flame, until the contents commence to boil. After this, the flame is so regulated that 110 ccs. of distillate pass over in 30 minutes, the rate of the distillation being kept as near as possible to these figures. The distillate should be received into a flask, accurately marked at 110 ccs.

*Titration of the Volatile Fatty Acids.*—The 110 ccs. of distillate, after thorough shaking, are filtered through a dry filter-paper; 100 ccs. of the clear filtrate are then measured into a flask of about 250 ccs. capacity, and 0.5 cc. of phenolphthalein solution (1 gramme in 100 ccs. of alcohol) added.

$\frac{N}{10}$  barium hydroxide solution is run into the fatty acids, until the red coloration, which remains permanently for 2 or 3 minutes, is produced. The number of ccs. of  $\frac{N}{10}$  barium hydroxide solution, required, should be increased by one-tenth, in order to allow for the 10 ccs. of acid distillate not used.

#### *Other Methods.*

The method, described, is essentially that devised by Leffman and Beam \* and is based on the Reichert-Meissl method.

Other methods, official in various countries, are similar in the main points to *Method I*.

Wollny,† in order to obviate errors arising from absorption of carbon dioxide, from variation in the size, shape and

\* Leffman and Beam, *The Analyst*, 1891, xvi., 153; 1896, xxi., 251.

† Wollny, *The Analyst*, 1887, xii., 203; and *Milch Zeit.*, 1887, Nos. 32–35.

nature of the apparatus, and from the differing strengths of solutions employed, has made certain conditions under which the estimation should be made. This has been adopted, in its essential features, by the A.O.A.C.

#### IODINE VALUE.

The principle underlying the iodine and bromine values of fats and oils has already been given in Chapter XXV., and the determination of the amount of iodine or bromine, fixed by a given acid or mixture of acids, affords useful information as to the nature of the fatty acids present.

The absorption of free iodine by fats and oils is slower than the absorption of bromine, but the following methods, described, will be found to give accurate and consistent results in the shortest possible time.

#### Method I.

The first method, which was devised by Hübl,\* is as follows :—

*Solutions required.*—(1) An alcoholic solution of mercuric chloride is prepared by dissolving 60 grammes of corrosive sublimate or mercuric chloride in 1 litre of 95 per cent. alcohol. The solution is filtered and placed in a well-stoppered bottle.

(2) An alcoholic solution of iodine is prepared by dissolving 50 grammes of iodine in 1 litre of 95 per cent. alcohol.

(3) A sodium thiosulphate solution (about 24 grammes to the litre) is standardised against pure, sublimed iodine or potassium bichromate.

Before determining the iodine values of fats or oils, equal volumes of the first two solutions are taken and thoroughly mixed in a well-stoppered bottle, where they should be allowed to remain for at least twenty-four hours, in order that any iodine-absorbing impurities may be eliminated.

\* Hübl, *Dinglers Polyt.*, 1884, ccliii., 284.



From 0.8 to 1.0 gramme of a solid fat, such as cacao butter, or up to 0.5 gramme of drying or non-drying, oils is weighed out and dissolved in 10 ccs. of pure chloroform, which should not contain any iodine-absorbing impurity. To the chloroform solution, are added 30 to 40 ccs. of the mixed iodine and mercuric chloride solution, more being introduced, if, on standing for two to three hours, the brown-coloured mixture lightens in colour to any extent.

A large excess of free iodine, about equal to the amount absorbed, should remain in the solution undergoing the treatment after the fat has fixed all the iodine of which it is capable.

The mixed solution should be allowed to remain in close-stoppered bottles in the dark for several hours, preferably from twelve to twenty-four hours, when the iodine absorption should be complete.

When the process is complete, from 10 to 15 ccs. of a 10 per cent. solution of potassium iodide are added with about 150 ccs. of water, and the excess of iodine, present, titrated with the standardised solution of sodium thiosulphate, a little starch paste being used as indicator towards the end of the titration.

The difference between the iodine found in the blank and in the real determination will give the amount of iodine absorbed, which, calculated on 100 grammes of fat, will give the iodine value, required.

As the mixed solution of iodine and mercuric chloride varies in strength from day to day and as the chloroform may contain certain quantities of iodine-absorbing impurities, it is most necessary to carry out a blank experiment side by side with the actual determination. The same quantities of solution and the same operations, as were involved in the actual determination, should be employed for the blank experiment, with the exception that, in the latter case, no fat is added.

The iodine value is always estimated as the amount of iodine absorbed by 100 parts of fat.

The actual products, formed during the Hübl process of estimating the iodine value, have been the matter for considerable discussion. From the researches of Hübl, Ephraim,\* Wijs,† Lewkowitsch,‡ and others, it would appear conclusive that, during the mixing of the iodine and mercuric chloride solutions, a certain proportion of mercuric iodide is formed with, possibly, a small quantity of iodine monochloride. It is probable, therefore, that the fatty halogen compound, eventually formed, is not a simple iodine addition product, but one containing both iodine and chlorine.

#### Method II.

A modified method, suggested by Wijs, has considerably lessened the time taken for absorption, use being made of the action of water on iodine chloride to form hypoiodous acid, which, he considers, to be the chief agent in accelerating the absorption.

Wijs prepared his iodine chloride by dissolving 13 grammes of iodine in a litre of glacial acetic acid and by introducing chlorine, until the amount of thiosulphate, required by the solution, was doubled. With a little practice, the correct point can be detected by the change in colour of the solution. The solution, so prepared, is practically an acetic acid solution of iodine chloride and is fairly stable. This solution is used in the same way as Hübl's mixed solutions, with the advantage that the time of absorption is greatly reduced.

#### Other Methods.

Other methods have been devised by Welmans§ and Waller|| and can be found in the original papers given in the

\* Ephraim, *Zeitsch. angew. Chem.*, 1895, 254.

† Wijs, *ibid.*, 1898, 291.

‡ Lewkowitsch, *The Analyst*, 1899, xxiv., 257.

§ Welmans, *Jour. Soc. Chem. Ind.*, 1900, xix., 694.

|| Waller, *The Analyst*, 1895, xx., 280.

footnotes, or in the works on oils, fats and waxes by Lewkowsch and Wright.

#### **Cacao Butter Substitutes: Special Tests.**

In Table LXXX. will be found physical and chemical constants for cacao butter and for those fats and oils with which it is likely to be adulterated. There are also included, in the same table, constants for those fats and oils which may be found in conjunction with cacao butter in the fat extracted by a solvent from commercial chocolates other than plain, such as milk, nut and other fancy chocolates.

The determination of fat-constants, the values for which are dependent upon the composition of the fat, affords some indication of the nature and extent of adulteration. Thus, a high iodine value suggests the presence of certain foreign vegetable oils, such as sesame, arachis or almond. A low saponification value will suggest beeswax or paraffin wax. A low iodine value will be found if coconut oil or stearin has been substituted for cacao butter.

In general, the vegetable oils increase the iodine absorption value and lower the melting-point of the insoluble fatty acids, obtained from the mixed fats.

Cacao butter may show a high acid value, if the fat has been extracted from shells (Dutch HA butter) or unduly exposed to the oxidising influence of air and light. The acid value, given by Matthes and Rohdlich,\* for cacao butter is 1.1 to 1.95.

There have been many researches upon the detection of cacao butter substitutes in chocolate. Coconut fat or stearin, after a great part of the olein has been removed, is especially suitable as a cacao butter substitute. Wauters† suggests the following method for the detection of coconut fat in cacao butter.

\* Matthes and Rohdlich, *Ber.*, 1908.

† Wauters, *The Analyst*, 1901, xxvi., 128 and 292.

Five grammes of the extracted fat (if from chocolate) is saponified, and the soap dissolved in 150 ccs. of boiling water; 50 ccs. of 5 per cent. sulphuric acid solution are then added, and the whole is distilled, so that 100 ccs. pass over in thirty to thirty-five minutes. After the first distillation, another 100 ccs. of water are added, and the distillation repeated. The two distillates are separately filtered, and 50 ccs. of each filtrate titrated against a  $\frac{N}{10}$  solution of caustic soda. The filtrates are washed with 50 ccs. of ethyl alcohol, and the washings mixed with the 50 ccs. of the filtrate and again titrated.

By this method, which is really an extension of that for estimation of the Reichert-Meissl value, Wauters obtained the following results, the figures being expressed as cubic centimetres of  $\frac{N}{10}$  caustic soda solution required for neutralisation :—

	Soluble volatile acids			Insoluble volatile acids		
	1st.	2nd.	Total.	1st.	2nd.	Total.
Coconut oil	7.1	4.3	11.4	7.85	7.55	15.4
Cacao butter	0.1	—	0.1	0.25	0.15	0.4

Sachs \* has examined many exotic vegetable fats, which have been used as substitutes for cacao butter. Dika or Gaboon fat, Tankawang fat or Borneo tallow and Illipé fat have been examined by this author, who gives also the constants he obtained for them. The same author has examined the stearin obtained from coconut and palm nut fats. He suggests the admixture of 75 per cent. coconut stearin with 25 per cent. Japan wax for a good cacao butter substitute. This mixture gives the following constants :—Melting-point, 34° to 35.5° C.; iodine value, 7.8; saponification value, 237; Reichert-Meissl value, 5.5. Other mixtures,

\* O. Sachs, *The Analyst*, 1906, 123.

recommended by Sachs, are two-thirds palm nut stearin with one-third coconut stearin, and, again, 40 per cent. Tankawang fat with 60 per cent. coconut stearin.

A new adulterant of cacao butter has been mentioned, though not identified.\* The fatty acids from the adulterated sample were brownish yellow and of coarse, granular structure, whilst the fatty acids from cacao butter are light yellow in colour and solidify in fine crystals. The critical temperature of solution in glacial acetic acid (5 ccs. of molten fat in 5 ccs. of acetic acid) of the adulterated sample was  $35^{\circ}$  C., that for pure cacao butter being  $76^{\circ}$ — $80^{\circ}$  C. Crystallisation, from ether-alcohol solution at  $10^{\circ}$  C., resulted in the production of an oily substance in half an hour, which, on recrystallisation from the same solvent, melted at  $30.7^{\circ}$ — $40^{\circ}$  C. Under similar conditions, pure cacao butter gave directly white crystals melting at  $46^{\circ}$ — $52^{\circ}$  C., and, after recrystallisation,  $56^{\circ}$ — $57.4^{\circ}$  C.

Bolton and Revis † have recently devised a modified Halphen test for the detection of so-called "green butters" when used as adulterants of cacao butter. The method is not intended to supersede any of the existing estimations of chemical and physical constants, but to supplement them in those cases where "green butters" are suspected. The adulterants under consideration conduct themselves like cacao butter towards Björklund's test and other solubility tests, so readily, that they do not admit of differentiation.

Halphen ‡ described his test substantially as follows:—One gramme of the absolutely clear, filtered fat is dissolved in 2 ccs. of carbon tetrachloride: to 2 ccs. of this mixture, is added a solution of bromine in carbon tetrachloride (made by adding bromine to an equal volume of carbon tetrachloride) drop by drop, until the colour of the bromine is just

\* *Chim. Rev. Fett. Ind.*, 1914, xxi., 47 and 74.

† E. R. Bolton and C. Revis, *The Analyst*, xxxviii., 201.

‡ Halphen, *Jour. Pharm. Chim.*, 1908, xxviii., 345.

permanent. To the mixture, are added 3 ccs. of petroleum, (S.G., 0.700) and the tube stoppered and allowed to stand for twenty-four hours at ordinary room temperature. Halphen states that, under these circumstances, a solution of cacao butter remains perfectly clear; whilst "green butters" give a flocculent precipitate.

Bolton and Revis found that several "green butters," examined, gave practically no precipitate, and that certainly 5 per cent. of added "green butter" could not always be detected as Halphen had stated. It was observed, however, that, on adding the bromine solution to the fat solution, turbidity always occurred when pure cacao butter was present, but that solutions of fat, adulterated with "green butters," and especially solutions of "green butters" alone, remained quite clear. The turbidity, moreover, was not dissolved or dissipated by petroleum ether, as it was by Halphen's petroleum. The authors claim that, with the modified test, described below, "green butters" can be differentiated from cacao butter, and that 10 per cent. of "green butter," added to cacao butter, can be detected. Bolton and Revis' test is as follows:—

One gramme of the clear, filtered fat is dissolved in 2 ccs. of a mixture of equal parts of carbon tetrachloride and petroleum ether (distilling below 40° C.). Two ccs. of this solution are placed in a test-tube about 6 inches long and  $\frac{1}{4}$  inch in diameter. The tube is cooled in water, and the solution of bromine in tetrachloride (prepared as in Halphen's test) added drop by drop, with constant shaking, until the colour of the bromine is permanent. More than one drop excess of the bromine solution should be avoided. The tube is corked and allowed to stand. If, after the expiration of fifteen minutes, the solution is perfectly clear, cacao butter is not present or there is less than 10 per cent. If the solution shows turbidity, cacao butter is indicated, except if Gutta serena fat is present when the turbidity is different and can be

readily distinguished by a method that the author provides. It is suggested that the degrees of turbidity enable the modified Halphen's test to be quantitative.

TABLE LXXX.—Physical and Chemical Constants of Cocoa Butter, etc.

Fat or oil.	Specific gravity. At ° C.	M. P. ° C.	M.P. of fatty acids. ° C.	Saponifica- tion value.	Reichert- Meissl value.	Iodine value.
Cacao butter . . .	0.964-0.974 at 15° 0.8577 at 98°	30-34	48-53	192-195	0.2-0.9	32-42
Possible adulterants:						
Bassia tallow . . .	0.9175 at 15°	25-42	39-5-45	187-194	0.5-0.8	54-68
(Mixture of Mohwah and Mahua butters)	0.8943-0.8961 at 100°	37.5	58-5	192.4-196	0.3-0.5	30-31
Borneo tallow . . .	0.9180-0.9217 at 15°	36-46	39-57	179-203	0.2	23-38
Chinese tallow . . .	0.867 at 100°	30-40	27-45	194.5	0.8-1.0	89-93
Cotton seed stearin . . .	0.911 at 50°	41-43	61-0	187-191.5	0.1-1.5	25-34
Gos butter . . .	0.8889 at 100°	29-40	51-55	201-221	1.5	43.5-56
(Kokum butter) . . .	0.902 at 40°/15°	43-51	42-5	154-161	1-4.2	48-85
Mafura tallow . . .	0.945-0.996 at 15°	27-43	48-50	200-205	0.8-1.9	53-68
Nutmeg butter . . .	0.9210-0.9245 at 15°	39	—	191	0.4	42
Palm oil . . .	0.8970 at 100°/100°	36-42	56	189-191	0.2-0.4	38-39
Phulwara butter . . .	0.915 at 15°	23-28	39.5-56	179-192	—	56-67
(Karité fat) . . .	0.8900 at 100°	20-28	24-25	246-262	6.6-8.4	8.2-9.5
Pinoy tallow . . .	0.9175 at 15°	29.3-29.5	28-1	252	3.4	4.0-4.5
(Malabar tallow) . . .	0.859 at 99.5-15.5°	23-30	21-28.5	243-255	5-6.8	10.5-17.5
Shea butter . . .	0.9239 at 15°	31.5-32	28.5-20.5	242	2.2	8
(Galam butter) . . .	0.8736 at 100°	42-50	41-47.5	196	0.3-0.5	36-42
Coconut oil . . .	0.8700 at 100°	30-44	37-47	195-203	0.2	47.5-34
Coconut stearin . . .	0.8731 at 99°/15.5°	47-49	34-36	196	0.3	33-50
Palm nut . . .	0.8700 at 100°	38-50	41-49	193-198	0.2	33-48
(Palm kernel oil) . . .	0.8750 at 15.5°	62-66	—	88-98	—	8.5-11.5
Palm nut stearin . . .	0.8626 at 98°-100°	36.7-38.3	—	—	—	3.9-4.0
Beef fat . . .	0.8600 at 98°-100°					
Lard . . .	0.937-0.953 at 15.5°					
Mutton fat . . .	0.925-0.940 at 15.5°					
Tallow . . .	0.959-0.970 at 15.5°					
Beeswax (normal) . . .	0.962-0.966 at 15.5°					
Paraffin wax . . .	0.842-0.140 at 15.5° (according to melting- point).					
Possibly present, due to addition of nuts, milk, etc.:		Solidification point.				
Almond oil . . .	0.914-0.920 at 15.5°	- 10 to - 20	13-14	186-192	0.5	93-100
Arachis (earth nut) oil . . .	0.911-0.926 at 15.5°	+ 3 to + 10	27-30	186-194	0.5	83-101
Hazelnut oil . . .	0.919-0.917 at 15.5°	- 10 to - 20	26-25	191-197	0.9-1.0	83-90
Pine-nut oil . . .	0.9215-0.9250 at 15.5°	- 18 to - 20	16-19	191-193	—	106-120
Walnut oil . . .	0.9240-0.9268 at 15.5°	- 12 to - 24	15-20	190-197	0.0	139-148
Melting- point.						
Butter fat . . .	0.909-0.913 at 38.5°	28-36	38-42	215.8-241.1	21.0-33.4	28-42

## CHAPTER XXX

METHODS OF ANALYSIS: MOISTURE—MINERAL MATTER (SOLUBLE, INSOLUBLE, ALKALINITY OF)—COLD-WATER EXTRACT—FIBRE (CRUDE FIBRE, CELLULOSE, PENTOSANS)—CACAO-RED—XANTHINE BASES (THEOBROMINE, CAFFEINE)—ORGANIC ACIDS (TARTARIC, ACETIC, OXALIC)

### Moisture.

THE estimation of moisture is of only minor importance to the analyst, though it is desirable for the manufacturer to keep this value as low as possible, seeing that mouldiness and general deterioration of quality result from excess of moisture in cacao and cocoa preparations.

### Method.

From 2 to 5 grammes of the powdered sample are distributed evenly over the bottom of a platinum dish, previously heated, cooled and weighed. The dish and its contents are placed in a steam-heated oven and maintained at 100° C., until, after frequent weighings, no further loss in weight occurs. The loss in weight may be attributed to water, lost at 100° C., the quantity of theobromine and other volatile matters, which escape at this temperature, being negligible.

It is eminently desirable that standard methods should be adopted for all estimations. In the case of determination of moisture, great variations may occur, according to the method adopted.\* Campbell† has shown some figures, confirming this, which are of interest. (See next page.)

\* R. Whympster, "Conditions that govern Staleness in Bread," 1918.

† H. H. Campbell, *Jour. Soc. Chem. Ind.*, xxxii., 69. See also *Ann. des Pâtisf.*, 1911, iv., 417.



*Percentage of Moisture in Sweetened Cocoa.*

Carbide method	Vacuum without heat	Vacuum at 98°	Air-oven at 100° C.	Differences.		
I	II	III	IV.	I. & II.	I. & III	I. & IV.
4.29	4.29	3.54	3.92			
4.38	4.34	3.58	4.08			
<i>Mean</i>						
4.33	4.32	3.56	4.00	+ 0.01	+ 0.077	+ 0.33

Method IV. is that adopted, as official, in France, the cocoa being heated in an air-oven at 100° to 105° C. for 6 hours.

**Mineral Matter or Ash.***Method.*

The quantity of sample, previously employed for moisture determination, is incinerated, firstly, over a naked gas flame, until the fat and other easily combustible matter is thoroughly charred, and, then, in a muffle furnace, until no further loss in weight occurs. The residue represents the mineral matter or ash.

If no addition of foreign mineral matter in the form of husk, alkali, etc., has been made, the amount of ash, found, is a direct indication of the quantity of cacao matter present in the sample.

*(a) SOLUBLE AND INSOLUBLE ASH.**Method.*

About 20 to 30 ccs. of distilled water are added to the mineral matter in a platinum dish, and the water brought to the boil. The solution is then filtered through a clean filter-paper, and the residue thoroughly washed with small quantities of hot water, until the filtrate amounts to about 80 ccs. The filtrate is evaporated in a clean, weighed platinum dish and heated in a muffle furnace. The increase in weight will be the soluble ash.

The filter-paper and residue are dried in a steam-oven and incinerated in a second platinum dish, previously weighed. The increase in weight, after allowing for the amount of ash contained in the filter-paper (usually about 0.001 per cent.), will be the insoluble ash.

The amount of insoluble is always higher than the soluble ash in pure cacao mass, being usually about 66 per cent. against 34 per cent. of the total ash, respectively. The roasted germ shows a higher soluble ash, Bordas finding 57 per cent. against 43 per cent. of insoluble ash. This author gives the same figures, reversed, for roasted shells, namely, 43 per cent. for soluble and 57 per cent. for insoluble ash.

Stutzer\* has shown, in a clear manner, how the soluble ash is increased by the addition of alkaline carbonates to cocoa preparations. He has stated that the ash from pure cocoa preparations does not effervesce with dilute mineral acids, and that it is soluble in water to the extent of one-third of its total weight.

Booth† has pointed out the importance of determining the ratio of soluble to total ash as confirmatory evidence of the addition of ochre and other mineral colouring matter. If the soluble ash is more than half that of the total, the addition of husk or added alkali is probable.

#### (b) ASH OR MINERAL MATTER INSOLUBLE IN ACID.

##### Method.

Two grammes of the sample are incinerated, as previously described, and boiled with 25 ccs. of 10 per cent. hydrochloric acid solution for 5 minutes.

The insoluble matter is collected and estimated by filtering the solution through a Gooch crucible, washing

\* A. Stutzer, *Zeitsch. angew. Chem.*, 1892, 510.

† N. P. Booth, *The Analyst*, 1900, xxxiv., 141.

the residue with hot water, igniting and weighing. The residue, obtained in this manner, is almost entirely composed of silica. The estimation of the ash insoluble in mineral acids may be of great value, as the figure is considerably higher in the husk than in the nibs of cacao.

(c) ALKALINITY OF THE ASH.

The official method of determining alkalinity of ash, adopted by the A.O.A.C.,\* is as follows:—

*Method.*

The entire ash, obtained by incinerating 2 grammes of the sample, is treated with excess of  $\frac{N}{10}$  hydrochloric acid solution (usually from 10 to 15 ccs.) in a platinum dish.

The solution is brought to the boiling-point by heating the platinum dish and contents over a Bunsen flame. The solution is then cooled, and the excess of hydrochloric acid titrated with  $\frac{N}{10}$  sodium hydroxide solution. The alkalinity is expressed as the number of ccs. of  $\frac{N}{10}$  acid required to neutralise the ash, obtained from 1 gramme of the sample.

In this work, the alkalinity has been usually expressed as potassium oxide ( $K_2O$ ), this being the method adopted by modern observers.

It is evident that the addition of alkalis, such as hydroxides and carbonates of sodium and potassium, in the preparation of cocoa or chocolate, will lead to the increased alkalinity of the ash, found.

Bordas has given figures showing the increase in potassium, estimated as oxide ( $K_2O$ ), found, by the addition

\* U.S. Dept. of Agriculture, 1907, Bull. 107.

of potassium carbonate or hydroxide, in certain commercial cocoas, examined. His results are tabulated below :—

Kind of cocoa	Salts of potassium in 100 parts of dry, defatted cocoa.	
	As $K_2O$ .	As $K_2CO_3$ .
Ordinary cocoas (mean)	2.54	3.87
Cocoa treated with alkali I.	4.82	7.07
Cocoa treated with alkali II.	4.86	7.12
Cocoa treated with alkali III.	5.02	7.36
Cocoa treated with alkali IV.	6.41	9.41
Cocoa treated with alkali V.	5.74	8.42

Bordas has stated that the addition of potassium salts renders the cocoas strongly alkaline, whilst ordinary cocoas are slightly acid in reaction.

Farnsteiner has pointed out that, if the ash obtained from 100 grammes of a sample of cocoa requires more than 15 ccs. of N acid for its neutralisation, and, at the same time, the ash insoluble in water is more than 60 per cent. of the total ash, the sample has been treated with magnesium carbonate. The same author has stated that the addition of ammonium carbonate can be detected by heating the cocoa with water and magnesium oxide in a flask, in the neck of which is suspended a piece of faintly red litmus paper. The ammonia, liberated by this treatment, will turn the red litmus paper blue. The quantity of ammonia, present, can be estimated by distilling into a standard  $\frac{N}{10}$  acid solution and by titrating the excess of acid remaining. Of this method we are extremely doubtful, since a large proportion of the volatile alkali must have been driven off by heat during the processes of manufacture.

In Baker and Hulton's paper, to which frequent reference has been made, will be found a summarised statement both on the ash soluble in water and the alkalinity of the soluble ash. We cannot but agree that "It is unfortunate that in

the figures recorded for the estimation (the latter) there has been so much ambiguity as to the method employed, the expression of 'alkalinity of ash', being often used without stating either the method of estimating or whether the result obtained is from the water-soluble ash or the total ash." These authors record figures, which we have already given for "the ash soluble in water," as varying between 72 and 25 per cent. of the total ash, though a figure of 35 per cent. has been found to be normal for both shell and nib of an average cacao. The same authors state that the soluble ash of an alkalisied cocoa is invariably well over 50 per cent., being often 60 and 70 per cent. of the total ash.

With regard to the alkalinity of the ash, Bakér and Hulton admit to the difficulty of determining whether alkalisiation has taken place by mere estimation of the alkalinity of the water-soluble ash, both owing to the different methods adopted for estimating alkalinity and because both soluble and insoluble non-volatile alkalis, as well as volatile alkalis, dissipated on the application of heat, may be used. It is for this reason that we have collected the more comparable results together in earlier pages.

#### GENERAL METHOD FOR DETERMINING ASH, ETC.

Purvis and Hodgson\* have given the following method for determining ash—its alkalinity and solubility.

##### *Determination of the Ash.*

Five grammes of the sample are placed in a platinum dish and carefully ignited over a Bunsen flame, until a white residue is obtained; the weight of this residue multiplied by 20 gives the percentage of ash.

\* J. E. Purvis and T. R. Hodgson, "Chemical Examination of Water, Sewage and Foods," 1914.

*Example.*—The residue obtained from 5 grammes of a sample of cocoa was 0.775 gramme, then  $0.775 \times 20 = 1.55$  per cent. of ash.

*Estimation of the Alkalinity of the Ash.*

The ash obtained above, is dissolved in water and washed into a beaker; a few drops of methyl orange are added, and the contents of the beaker are titrated with  $\frac{N}{10}$  sulphuric acid from a burette, until a permanent pink colour is obtained. Each cc. of  $\frac{N}{10}$  sulphuric acid used 0.0069 gramme of alkalinity, calculated as  $K_2CO_3$ .

*Example.*—The ash of a cocoa required 16.5 ccs. of  $\frac{N}{10}$  sulphuric acid to produce a permanent pink colour; then  $16.5 \times 0.0069 \times 20 = 2.28$  per cent. of alkalinity, calculated as  $K_2CO_3$ .

*Estimation of Soluble Ash.*

The residue from the cold-water solids is gently ignited over a Bunsen flame, and the ash weighed; the weight is multiplied by 500 to give the percentage; this should not be less than 2 per cent.

*Example.*—The ash of a sample of cocoa, obtained as described, weighed 0.005 gramme; then  $0.005 \times 500 = 2.5$  per cent.

Probably, the most recent important contribution to the detection of alkalisied cocoas is that given by the analyst Rocques,\* who, after Arpin's work already quoted, devoted himself to ascertain suitable methods for determining the quantitative valuation of alkalisiation. He estimated the total ash, the ash soluble and insoluble in water, the alkalinity of the soluble ash, and  $P_2O_5$  in the soluble ash. The

\* Rocques, *Ann. des Falsif.*, January, February, 1917, xcix., c. 14.

following is the method that he suggested should be official:—  
 First estimate the percentage of water ( $H$ ) and fatty matter ( $G$ ) in the sample, then, by taking  $\frac{500}{100 - (H + G)}$  grammes of the sample, an amount equal to 5 grammes of dry, defatted cocoa is used. The sample is incinerated at red heat in a platinum capsule, and, when free from carbon, the ash is moistened with a little water and a trace of pure ammonium carbonate and evaporated. The dish is again brought to a red heat for a short time, cooled and weighed. The weight of ash, obtained, multiplied by 20 gives total ash per cent. of dry, defatted cocoa.

The soluble ash is estimated by treating the total ash with a little boiling water and by stirring for a few minutes. The liquid is decanted through a filter into a 200 ccs. flask, and the ash, in the dish, treated again with boiling water 5 or 6 times. The filter-paper and contents are washed and replaced in the capsule, calcined to redness and weighed for the insoluble ash. The difference, between the total and insoluble ash, is soluble ash. The flask, containing the filtrate of the washings, is cooled, a drop of tropeoline solution added, and the whole titrated with  $\frac{N}{2}$  sulphuric acid.

The number of ccs. of acid, used to neutralise the solution, multiplied by 0.69 gives the alkalinity per cent. of the dry, defatted cocoa, expressed as  $K_2CO_3$ . This alkalinity, in the presence of tropeoline, corresponds to  $CO_2 + \frac{1}{3} P_2O_5$ , and, in order to estimate the alkalinity corresponding to  $CO_2$  alone, an excess of about 5 ccs. of  $\frac{N}{2}$  acid are added to make a total volume of 10, 15 or 20 ccs. The solution is heated to boiling for 10 minutes, cooled and titrated with  $\frac{N}{2}$  caustic soda solution in the presence of phenolphthalein. The acid and alkaline solutions must be very carefully

adjusted. Then, if  $N$  = number of ccs. of  $\frac{N}{2}$  sulphuric acid, added, and  $n$  = the number of ccs. of  $\frac{N}{2}$  caustic soda, used, the alkalinity as  $K_2CO_3$  will be  $(N - n) \times 0.69$ . The amount of  $P_2O_5$  is estimated in the same solution by adding a trace of pure ammonium chloride and, then, in succession, 20 ccs. of ammonium citrate, 50 ccs. of ammonia and 10 ccs. of a magnesia solution. The weight of magnesium phosphate multiplied by 12.8 gives  $P_2O_5$  per cent. of dry, defatted cocoa.

A series of graphs are shown in Rocques' paper, and, whilst the ratio, insoluble ash : soluble ash, is in the neighbourhood of 2 for pure cocoa, it becomes 1 and lower for alkalisated cocoas.

In order to determine the proportion of alkali, added to cocoa, it is necessary to establish the limits within which pure cocoas vary, and the author recommended that those to whom pure cocoas are readily available should establish these points.

#### • Cold Water Extract : Cacao Matter Soluble and Insoluble in Cold Water.

The amount of matter soluble in cold water is, according to Booth, about 24 per cent. of the fat-free nib. Bordas, however, gives 9.67 per cent. for the same estimation.

• We have obtained figures ranging from 9 per cent. to 12.5 per cent. for the whole nib, results which are in accordance with those obtained by the first author.

The addition of sugar, alkaline salts, etc., will considerably affect this value. Thus, in chocolate, the cold-water extract may rise to 70 per cent.

Bordas has made use of the matter insoluble in cold water for estimating the amount of defatting that a cocoa has undergone. Booth, on the other hand, has used the cold-



water extract for estimating the amount of cacao matter present in a sample, on the assumption that cocoa nibs contain 12 per cent. of soluble extractive.

#### Method.

A simple method for the estimation of the cold-water extract is as follows :—

Two grammes of the sample are placed in a 100 cc. flask and shaken with 50 to 60 ccs. of water. The solution is then made up to 100 ccs. and left standing overnight. Twenty-five ccs. of this solution are filtered off, evaporated, dried and weighed.

More rapid and consistent results will be obtained if the material is defatted before extracting.

Purvis and Hodgson's method is slightly different :—

Thoroughly mix 5 grammes of the cocoa with water in a glass mortar : wash the mixture into a 250 cc. flask, and make up to the mark ; the mixture is allowed to stand for twenty-four hours with intermittent shaking, and it is then filtered ; 10 ccs. of the filtrate are evaporated to dryness in a weighed, glass dish on the water-bath, dried in the water-oven and weighed ; the weight of the residue, obtained, multiplied by 500 gives the percentage of the water-extract. Should this percentage exceed 18, then added sugar is present.

*Examples.* The dry residue of a sample of cocoa, obtained as above, was 0.024 gramme ; then  $0.024 \times 500 = 12$  per cent. of water-extract. The sample, therefore, does not contain added sugar. Another sample of a residue of 0.066 gramme ; then  $0.066 \times 500 = 33$  per cent. of water-extract ; the sample contains sugar.

#### Fibre.

The determination of fibre, though simple in itself, is not readily interpreted:

Firstly, the complex nature of fibre makes it difficult to say what is precisely obtained by any given treatment, and,

secondly, the word "fibre" conveys to the mind the cellulose tissue of plant life, whilst it is not cellulose, alone, which has been estimated when the value for fibre is given.

From the most recent researches, it has been shown that the cell-walls of plant life are composed, for the most part, of compounds of sugars (of the hexoses and pentoses). The outer cellular structure of seeds contains cellulose and these same sugar compounds, intermingled with various organic bodies, colouring matters, tannin, etc.

The estimation of crude fibre, therefore, may be the estimation of cellulose, or a mixture of cellulose and other bodies, according to the treatment to which the material is subjected.

The amount of crude fibre, found, is greater in the outer shells or husks than in the kernel or nibs of cacao, and, whether it is the direct estimation of the "crude fibre" of Filsinger, the determination of the cellulose of Weender, or the indirect estimation of fibre by determination of the pentosan-content of the cacao, as carried out by Tollens and his pupils, or by König, the values, obtained, are an indication of the extent of adulteration of cocoa preparations with husk, so long as the experimenter works always with the same method, standardised by himself against pure husk and cacao mass and known mixtures of the two. Various other methods and their interpretations have been left for a later chapter.

#### (a) CRUDE FIBRE.

##### *Method I.*

Weender's process, modified by König, for estimating the amount of crude fibre (chiefly as cellulose) consists in treating 3 grammes of the defatted sample with 200 ccs. of glycerol (S.G. 1.23), containing 4 grammes of concentrated sulphuric acid, under a pressure of 3 atmospheres for one hour. The solution is then filtered, whilst hot, through an asbestos filter,

washed successively with hot water, alcohol and ether, and the residue dried and weighed.

The residue is then incinerated, and the ash weighed. The difference between the two weighings gives the amount of ash-free crude fibre present in the sample.

#### *Method II.*

An alternative method for the estimation of crude fibre is as follows :—

Two grammes of the sample are freed from fat by extraction with ether, and the residue boiled for half-an-hour under a reflux condenser with 200 ccs. of water and 2.5 ccs. of sulphuric acid. The liquid is filtered through linen, and the residue, after filtration, is thoroughly washed with hot water and, subsequently, boiled with 200 ccs. of 1.25 per cent. caustic soda solution. The residue, undissolved, is filtered off, washed successively with hot water, alcohol and ether, dried at 100° C. and weighed.

In cocoa free from husk, the value for crude fibre, obtained in this manner, will amount to 2 per cent. or 3 per cent., but will exceed this limit in proportion to the amount of husk present.

#### *Method III.—Direct Estimation of Husk.*

As the determination of crude fibre is practically a measure of the amount of husk present in a cocoa, Goske's \* method of direct estimation of husk, based upon the difference between the specific gravity of cocoa husk and that of ordinary cocoa powder, should be mentioned here.

Dubois and Lott † have recently studied this method and state it to be of only approximate accuracy.

Goske recommends that 1 gramme of dry fat-free cocoa be mixed with a calcium chloride solution (of specific gravity

\* A. Goske, *Zeitsch. Nahr. Genussm.*, 1910, xix., 154.

† Dubois and Lott, *Jour. Ind. Eng. Chem.*, 1911, iii., 251.

=1.535 at 30° C., prepared by dissolving 107.1 grammes of calcium chloride in 100 ccs. of water), in a stoppered tube.

After thorough shaking, the stopper of the tube is removed, and the whole boiled for two minutes. While still hot, the tube is submitted to centrifugal action for six minutes. The top liquid portion is poured off, and the sediment collected on a weighed filter-paper, washed well with hot water, until the filtrate is free from chlorine, dried and weighed.

On the assumption that cacao husk yields 38.7 per cent. of dried sediment, this being the maximum figure found for husk, and that commercial cocoa powders yield 6 per cent., the percentage of added husk in a cocoa powder can be readily calculated. Allowance must be made for the fat removed.

An example given is that of a sample of cocoa, 1 gramme of which gave 0.0618 gramme of sediment. Using the figure 38.7, this amount is equivalent to 0.16 gramme of husk. The cocoa powder contained 18.4 per cent. of fat, hence an amount equivalent to 13 per cent. of husk was originally present. Allowing for the 6 per cent. contained by normal cocoa powders, the sample contained 7 per cent. of added husk.

It is a mistaken notion to imagine that any great degree of accuracy in the estimation of husk present in cocoas can be obtained by the "levigation" method, or by that elaborated, from Filsinger's\* original method, by Macara and others, so long as the experiments are made on cocoa powders that have been reduced to an extremely fine state of division. Baker and Hulton must have realised this; indeed, they admit to the weakness of the method, yet these two authors seem inclined to put more faith in "levigation" than in analytical methods, such as estimation of crude fibre and pentosans, either of which is a better indication of the amount of, and contamination with, husk, especially when taken in

\* Filsinger, *Zeitsch. öffent. Chem.*, 1899, 29.

conjunction with the figures obtained for the ash and the alkalinity of the ash. It is a pity, therefore, that Baker and Hulton, having the excellent opportunity of impressing upon the authorities the desirability of a standard method for estimating husk in cocoas, did not take greater advantage of their position and, at any rate, complete the comparison between the amount of husk, calculated from the crude fibre estimation and that calculated by the "levigation" method, and the estimation of nitrogen. We have occasion to refer to these results in a later chapter. The "levigation" method adopted by Baker and Hulton is as follows:—

"Ten grammes of the finely ground material to be analysed are extracted for twenty hours with ethyl ether in a Soxhlet apparatus. The extracted mass is exposed to the air to remove adhering ether, dried in the water-oven, well ground in a mortar, then stirred into a thin paste with water and transferred with washing to a 500 cc. cylinder. The volume in the cylinder is made up to 400 ccs., the whole inverted a few times to ensure admixture, and an arrangement inserted into the neck for blowing off the supernatant liquid. The leading tube should be turned up at the lower end to avoid any disturbance of the sediment. The contents of the cylinder are allowed to stand for fifteen minutes, and the liquid blown off. The volume is again made up to 400 ccs. with water, and the procedure above described repeated after ten minutes and again after two periods of five minutes' stand. Should the residue in the cylinder show the presence of much starch it should be finely ground in a mortar and again submitted to levigation. The residual sediment is transferred to a platinum basin, the water removed by evaporation, and the residue dried in the water-oven and weighed. The ash obtained from this residue is then deducted. We have adopted the figures for the sediment obtained from nib and shell as found by Bolton and Revis (*loc. cit.*)—namely, 3 per cent. for dry fat-free nib and

30 per cent. for dry fat-free shell. The percentage of shell in the original sample may be calculated from the formula:—

$$S = \left( \frac{100 M}{100 - (F + W)} \right) - 3 \times \frac{100 - (F + W)}{27},$$

where  $S$  = percentage of shell on sample,

$M$  = percentage of ash-free levigation sediment on sample,

$F$  = percentage of fat on sample.

$W$  = percentage of water on sample."

#### (b) CELLULOSE.

The method of estimating cellulose is practically the same as that for crude fibre. The amount of cellulose, found, must depend upon the severity of the treatment, and it is for this reason that so many variations in figures, given by different experimenters, are to be observed.

##### *Method I.*

From 1 to 5 grammes of sample are boiled with a dilute mineral acid, in order to render the starch completely soluble. The solution is filtered, and the residue thoroughly washed with hot water and, subsequently, warmed with a 1 per cent. solution of soda. The solution is filtered, and the residue washed successively with water and alcohol, dried at 100° C. and weighed. The weight, found, after a deduction has been made for any mineral matter which the residue might contain, may be considered to be cellulose and is a fairly constant quantity, amounting to about 4.9 per 100 parts of pure cacao mass, or 14.9 for 100 parts of cocoa matter insoluble in water.

Any addition of husk, germ or cacao waste tends to increase the cellulose and to decrease the starch-content of the cocoa.

*Method II.*

To obtain true cellulose, the following process of König is recommended :—

To determine cellulose, lignin and cutin in crude fibre, König\* digests the fibre in the cold with hydrogen peroxide in presence of ammonium hydroxide, the treatment being continued for a long time with successive additions of hydrogen peroxide, until the residue is colourless. This treatment oxidises the lignin and converts it into soluble products. The residue, consisting of cellulose and cutin, is treated with cuprammonium solution, to dissolve the cellulose, the cutin remaining unattacked. The liquid is filtered on a Gooch asbestos filter, and the cutin residue weighed; the cellulose is precipitated by alcohol and weighed, and the weight of crude fibre, less the weight of these two, is taken as lignin.

## (c) PENTOSANS.

As already described in a previous chapter, the fibrous material, comprising the cell-walls of plants, is composed chiefly of anhydrides of pentoses or sugars containing  $C_5$ . These anhydrides are called pentosans and yield, on hydrolysis, a pentose sugar xylose, arabinose, etc.

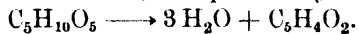
The quantitative determination of xylose and arabinose, respectively, is comparatively simple, but, in the vegetable kingdom, these sugars are in a condensed form and associated with hexoses and other sugars which complicate their direct determination.

It becomes necessary, therefore, to determine the pentosans, present, by the indirect methods of estimating the products which are formed from them by chemical action, and which are characteristic of the pentosans and not of the other sugars with which they may be associated.

Tollens and his pupils have been largely instrumental

\* König, *Zeitsch. Nahr. Genussm.*, 1906, xii., 385—395.

in perfecting processes by which the pentosans may be estimated, and which rest mainly on the quantitative determination of furfural, a decomposition product which is formed by heating the pentoses with dilute hydrochloric acid. The chemical explanation, which is really merely one of abstraction of water, is explained by the equation:—



#### Method I.

This method, which has been used with success by Hehner and Skertchley,\* Adan,† and others, and suggested by Tollens in conjunction with Gunther and de Chalmont, has been shown to have an efficiency of from 99.1 per cent. to 101.1 per cent. over a range of experiments, carried out by Adan.

Three to 4 grammes of the sample are placed in a Wurtz flask, of 250 ccs. capacity, with 100 ccs. of dilute hydrochloric acid of 1.06 specific gravity (equivalent to 12 per cent. HCl). The contents are then distilled, until 30 ccs. have passed over, when 30 ccs. of dilute acid are let in to the flask, to take the place of that portion which has distilled over. This process is continued, until about 300 ccs. of distillate have been collected. As the distillation proceeds, the furfural can be detected by allowing a drop of the distillate to fall on a filter-paper, which is then tested with a drop of dilute aniline acetate solution, containing a small proportion of sodium acetate. When a pink coloration is no longer produced, the distillation is stopped.

The distillate is made up to 400 ccs. with dilute hydrochloric acid (specific gravity 1.06). This solution is then, slowly neutralised with dry and finely powdered sodium carbonate and faintly acidified with acetic acid; 10 ccs. or more, if required, of an acetic acid solution of

\* O. Hehner and W. P. Skertchley, *The Analyst*, 1899, xxiv., 178.

† R. Adan, *Bull. Soc. Chim. Belg* 1907, xxi., 211.



phenylhydrazine (12 drops of phenylhydrazine to 7.5 ccs. of strong acetic acid) are added, and the solution constantly stirred.

The volume is then increased to 500 ccs. with constant stirring for half-an-hour. The precipitate is collected on an asbestos filter, washed thoroughly and quickly with water and dried in a current of warm air ( $50^{\circ}$  to  $60^{\circ}$  C.), under slightly reduced pressure.

The weight of precipitate multiplied by 0.516 will give the weight of precipitated or distilled furfural.

An error of about 0.25 per cent. may occur, if the amount of furfural, remaining in the solution and washings, has not been included.

#### Method II.

This method, suggested by Counciler, has been used by König with success and is similar in operation to *Method I.* up to the end of the distillation of the furfural.

The 400 ccs. of acid distillate are treated with a solution of pure phloroglucinol in dilute hydrochloric acid, and the mixture allowed to stand overnight. The precipitate is then collected on an asbestos filter, thoroughly washed and then dried just below  $100^{\circ}$  C. for several hours, till no further loss in weight occurs.

An efficiency of 99.5 per cent. to 101.2 per cent. was obtained, by this method, by Adam.

From Tollens' data, the amount of furfural can be calculated after the following corrections have been made:—

One gramme furfural produces 1.82 grammes phloroglucide for 0.1 gramme precipitated furfural.

One gramme furfural produces 1.92 grammes phloroglucide for 0.2 gramme precipitated furfural.

One gramme furfural produces 1.93 grammes phloroglucide for 0.3—0.5 gramme precipitated furfural, and, by interpolation, the intermediate figures can be calculated.

From the furfural, so found, a value 0.0104 is subtracted, and the remainder multiplied by 1.88, or its correction, will give the equivalent of pentosan.

The percentage of pentosan can thus be readily calculated.

As has already been stated, the results of this method show a satisfactory efficiency, when pure furfural has been used for test purposes, and it was recommended by the Committee of the International Congress of Applied Chemistry (1909) as an excellent criterion of the purity of cocoa powders with regard to their freedom from husk.

### Cacao-Red.

The pigment of cacao is an oxidation product of tannin and is not apparent in the fresh beans.

By the action of light and air, the cacao-red is developed in much the same way as the colouring matter of the oak and kola nut. The original pigment compounds have been shown to decrease during fermentation (*vide* Chapter IX., Table XIV.), as also have the astringent matters.

Schweitzer\* has shown that a glucoside, cacaonin, is decomposed by a diastatic ferment, with the production of sugar and cacao-red, during oxidation of the seed. The pigment, obtained in this way, is of a complex nature and consists of tannin and resin, besides the red-brown amorphous colouring matter to which the name of "phlobaphene" has been given.

The pure cacao-red is only of scientific interest, and the estimation of the colouring matter of cacao is seldom required.

It should be mentioned, here, that Ulrich† has devised a method for estimating the amount of added husk to cocoas, based on the statement that cacao-red is to be found in the

\* C. Schweitzer, *Pharm. Zeit.*, 1896, 389.

† C. Ulrich, *Arch. Pharm.*, 1911, cexlix., 524.

nibs and not in the husk of cacao. This process is discussed in a later chapter. (Chapter XXXII.)

### *Method.*

From 2 to 3 grammes of fat-free cocoa are made into a paste with hydrochloric acid and exhausted with 100 ccs. of absolute alcohol in a Soxhlet tube. The hydrochloric acid is fixed by placing in the flask, below, some silver oxide, so that any acid, extracted with the alcohol, is converted into insoluble silver chloride.

The alcoholic liquid, after cooling and filtration, is treated with alcoholic lead acetate solution, when a purple-black precipitate is formed.

The precipitate is collected on a filter-paper, washed well with hot water and transferred to a small flask with a small quantity of 70 per cent. alcohol.

The lead salt in suspension is then decomposed by sulphuretted hydrogen, the solution warmed, to get rid of the sulphuretted hydrogen, and then filtered. The lead sulphide will remain as residue, whilst an alcoholic solution of cacao-red passes through as filtrate. From the alcoholic solution, the pigment can be obtained as a solid by evaporation and, after drying, weighed.

An alcoholic solution of cacao-red, when viewed through a spectroscope, gives a diffuse band in the green, allowing the red, blue, and most of the yellow rays to be transmitted.\*

### **Xanthine Bases (Theobromine and Caffeine).**

The determinations of the alkaloids of cacao are of importance, principally due to the fact that cocoa preparations owe their stimulating property to their presence.

The methods, recommended for the estimation of the alkaloids, are numerous, but, unfortunately, they do not give consistent results.

\* Blyth, "Foods, their Composition and Analysis," 1909, 369.

The majority of the methods, suggested, are untrustworthy, especially those which rely upon the estimation of theobromine by sublimation. The most systematic results have been obtained by employing the methods described by Kunze\* and Dekker,† a modified combination of the two methods being made a provisional standard by the A.O.A.C.

*Method I. (Kunze's Method).*

Ten grammes of the sample are boiled for 20 minutes with 100 ccs. of 5 per cent. sulphuric acid. The solution is filtered, and the residue thoroughly washed with boiling water.

The warm filtrate is precipitated with excess of phosphomolybdic acid, and, after 24 hours' standing, the precipitate is collected on a filter-paper and washed with about 1 litre of 5 per cent. sulphuric acid.

The filter, containing the moist precipitate, is transferred to a large dish and treated with excess of cold baryta, after which carbon dioxide is passed in, to precipitate the barium hydroxide. The whole is then dried on a water-bath and extracted with chloroform. The chloroform is distilled off from the clear filtered extract, when a perfectly white deposit of the two alkaloids, theobromine and caffeine, is left.

The white residue is weighed, dissolved in ammonia and heated to boiling-point. Excess of silver nitrate is then added (11.3 parts of silver to 1 part of theobromine), and the boiling continued, until no further ammonia escapes and until the liquid is reduced to a few cubic centimetres.

An insoluble substitution product of theobromine ( $C_7H_7AgN_4O_2$ ) is obtained, while the caffeine remains, uncombined, in solution. This insoluble precipitate is filtered off, washed and weighed. The silver is estimated in the

\* W. E. Kunze, *Zeitsch. anal. Chem.*, 1894, xxxiii., 1.

† J. Dekker, *Rec. Trav. Chim.*, 1903, xxii., 143.

dried, theobromine-silver compound by ignition, or by dissolving in nitric acid and precipitating as chloride.

By either of these means, the quantity of theobromine, in combination with the silver, and originally present, can be readily determined. The difference between the weight of theobromine, found, and the total alkaloids will give the amount of caffeine, present.

*Method II. (Dekker's Method).*

This process, which has greater brevity in its favour, is capable of giving very accurate results.

Ten grammes of the powdered sample are heated with 5 grammes of magnesium oxide and 300 ccs. of water in a reflux condenser for 1 hour. After filtering and draining on a pump, the residue is again boiled with water for 15 minutes and drained. The mixed solutions are evaporated to dryness, and the resulting residue triturated with fine sand and boiled with 100 ccs. of chloroform. The clear chloroform solution is now filtered and evaporated to dryness, and the white residue, consisting of theobromine and caffeine, weighed.

If it is desired to estimate the two alkaloids separately, the dried residue is treated with 50 ccs. of benzol for 24 hours, by which means the caffeine is dissolved. The solution is evaporated to a volume of 25 ccs. and filtered. The residue, consisting of theobromine, is dried and weighed.

By this process, Dekker obtained results varying from 1.69 per cent. to 1.73 per cent. of mixed alkaloids in cocoa.

Welmans\* has obtained satisfactory results by a slight modification of Dekker's method, and Fromme† has verified the accuracy of the process. Katz‡ has also experimented with the method and has obtained consistent results.

\* P. Welmans, *Pharm. Zeit.*, 1902, xlvii., 858.

† J. Fromme, *Apoth. Zeit.*, 1904, xviii., 68.

‡ Katz, *Chem. Zeit.*, 1903, xxvii., 958.

Maupy,\* by a slightly different process, obtained results agreeing with Dekker's figures, namely, 1·3 per cent. to 1·6 per cent. of mixed alkaloids in roasted nibs, and 0·54 per cent. in chocolate containing 60 per cent. of sugar.

Kreutz,† by yet another method, has succeeded in obtaining consistent results, though, in all cases, his figures are somewhat higher than those obtained by Kunze's and Dekker's methods. Kreutz finds total alkaloids from 1·99 per cent. to 3·85 per cent. in the nibs, 1·99 per cent. to 2·23 per cent. in Van Houten's cocoa, and 2·63 per cent. to 2·92 per cent. in Suchard's cocoa.

The reason for the inconsistency among the results, obtained, is owing largely to the fact that a portion of the theobromine is combined as a glucoside and is not set free without treatment with an acid or other hydrolysing agent. Kreutz found that only half of the total theobromine is immediately capable of extraction by chloroform, the remainder becoming soluble only after the glucoside has been hydrolysed.

There are a number of other results and methods that should be recorded. Prochnow,‡ by his own method, obtained 0·578 to 1·380 of mixed alkaloids per cent. in roasted cacao husks; 1·460 to 1·812 per cent. in unroasted nibs and 1·536 to 1·880 per cent. in roasted nibs, thus confirming Knapp's findings of a slight increase after roasting. Camilla and Pertusi§ found traces of xanthine bases in cacao butter, by taking advantage of the ease with which these bases are oxidised to carbylamine, a readily identified substance, owing to its characteristic smell. Their method for estimation of the bases is somewhat similar to that of Prochnow and is briefly as follows:—

Ten grammes of material are boiled for thirty minutes

\* L. Maupy, *Jour. Pharm. Chim.*, 1893, v., 329.

† A. Kreutz, *Zeitsch. Nähr. Genussm.*, 1908, xvi., 579.

‡ A. Prochnow, *Arch. Pharm.*, 1909, ccxlvii., 698.

§ S. Camilla & C. Pertusi, *Ann. Lab. Chim. Cent. delle Gabelli*, 1912, vi., 611.

with 150 ccs. of water and 50 ccs. N sulphuric acid. The liquid is then diluted to 500 ccs., heated on a water-bath and filtered hot. The filtrate is cooled, and 250 ccs. of it neutralised with magnesium oxide, evaporated to about 80 ccs. and extracted in a special Soxhlet extractor, or by shaking in a separating-funnel with three times its volume of carbon tetrachloride. This solvent removes the caffeine, but a small quantity of the theobromine will remain in the aqueous layer. The carbon tetrachloride is evaporated to dryness after the addition of a little paraffin wax, and the residue washed repeatedly with small quantities of boiling water, slightly acidified with sulphuric acid. The filtered aqueous extracts are added to the original layer, and the whole mixed with 5 grammes of magnesium oxide and evaporated to dryness. The residue is powdered and extracted, four or five times, with successive portions (100 ccs.) of boiling chloroform for ten minutes each time. The mixed alkaloids should be perfectly white on evaporation of the chloroform, but, if further purification is required, an aqueous solution is made, treated with 1 per cent. solution of cold potassium permanganate and again extracted with chloroform.

Another similar method has been described by Savini.\*

#### Organic Acids (Acetic, Tartaric, and Oxalic Acids).

Schweitzer† has pointed out that the plant acids, malic and tartaric acids, occurring in cacao, are due to the decomposition of the glucoside cacaonin, which, under the action of a ferment, breaks up into sugar, malic and tartaric acids.

On referring to Chapter IX., Table XIV., it will be seen that a loss of both free and combined tartaric acid occurs and that an increase in free acetic acid is noted during the processes of fermentation and curing of the beans.

Professor Harrison, who conducted the latter research,

\* G. Savini, *Ann. Chim. Applic.*, 1916, vi., 247.

† C. Schweitzer, *Pharm. Zeit.*, 1898, 390.

has systematically studied the chemistry of cacao from the fresh beans through the various operations which render them fit for the market, and his results are not only rational but consistent over a number of experiments. • It is to be doubted whether Schweitzer has fully proved the accuracy of his statement, or if the production of tartaric acid, by the hydrolysis of the glucoside, is sufficiently large to be taken into account.

The increase in acetic acid during fermentation is incontestable, due to the fact that the alcohol, formed during the first period, is converted by *Mycoderma aceti* into acetic acid, which can be detected in the cured commercial beans by the vinegar-like smell, as well as by chemical analysis.

The determinations of tartaric and acetic acids are not of commercial importance, and the total acidity of an aqueous extract of cacao nibs, estimated in terms of  $K_2O$ , seldom exceeds 0.75 per cent. • • •

#### TARTARIC ACID (WEIGMANN'S METHOD).

From 5 to 10 grammes of the sample are treated with 50 ccs. of water and thoroughly shaken. The aqueous extract is neutralised with ammonia and treated with calcium chloride solution. The precipitate is filtered and dissolved in hydrochloric acid and, then, reprecipitated with caustic soda solution.

By this method, Leffman and Beam\* found from 4.84 per cent. to 5.82 per cent. of tartaric acid to be present in cacao. • • • These we consider to be very high figures, for the total tartaric acid found by us has not exceeded 1 per cent.

#### OXALIC ACID.

The older experimenters on cacao do not include oxalic acid among the constituents, though the presence of oxalic acid in vegetable matters has been recognised for a considerable time. •

\* Leffman and Beam, "Food Analysis," 1901, 275—282.



Gautier has given values of 0.375 per cent. oxalic acid in black tea, 0.043 per cent. in coffee, and 0.247 per cent. in stick rhubarb. The same author has found 0.45 per cent. of oxalic acid in cocoa powder.

A certain amount of interest is attached to the estimation of oxalic acid, in that Albahary \* has found that the acid is present mainly as insoluble calcium oxalate in ordinary cocoa powder, whilst treatment with alkali, in the preparation of "soluble" cocoa powder, brings about the decomposition of the calcium oxalate with the production of soluble alkaline oxalates.

In two samples of cocoa powder, marked *N* and *P*, respectively, the former treated with alkali and the latter an ordinary cocoa powder, Albahary obtained 0.3459 per cent. of oxalic acid, combined as soluble oxalate, out of a total of 0.3647 per cent. in sample *N*, and only 0.0159 per cent. of soluble oxalate in sample *P*, with 0.3763 per cent. combined with calcium.

The researches of Neubauer,† Schultzen,‡ Salkowski§ and others on the estimation of oxalic acid in cacao and cocoa preparations have been carefully examined by Albahary, who has proposed the adoption of the following method.

#### *Method.*

About 50 grammes of the sample, dried over sulphuric acid until of constant weight, are placed in a beaker with 50 ccs. of a 10 per cent. sodium carbonate solution. The beaker and contents are heated on a water-bath for one hour, the liquor evaporating being replaced by an equal amount of water. To the solution, are then added 50 ccs. of a solution containing 10 parts of magnesium chloride and 20 parts

\* J. M. Albahary, *Internat. Cong. App. Chem.*, 1909, viii. C, 175.

† Neubauer, *Zeitsch. anal. Chem.*, 1868, ii., 499; and vii., 225.

‡ Schultzen, "Reichert und Dubois Reymond's Arch.," 1868, 718.

§ Salkowski, *Zeitsch. phys. Chem.*, x., 120.

of ammonium chloride to the 100 ccs. A small quantity of animal black is added, and the liquid stirred whilst the solution continues to concentrate on the water-bath.

After an hour and when the volume has been reduced by about one-half, the solution is filtered on a pump without allowing it to get cold, and the insoluble residue thoroughly washed with boiling water, until the filtrate is quite neutral. The filtrate is again concentrated on the water-bath, and, after the addition of enough ammonia to render the solution strongly alkaline, the beaker and contents are left to cool for twelve hours. The solution is then filtered, and a slight excess of calcium chloride solution and enough acetic acid, to render the solution faintly acid, are added. The solution is again left to stand for twelve hours. All the calcium oxalate is thrown down in this manner, and, after collecting the precipitate on a filter, the oxalic acid is estimated by igniting the dried residue, weighing the oxide of calcium, so formed, and calculating the oxalic acid that it represents.

Girard, who has recently carried out researches on the oxalic acid-content of cacao and cocoa preparations, has given some valuable figures, a few of which have been shown in Chapter XXVI., Table LXXII.

## CHAPTER XXXI.

### METHODS OF ANALYSIS: REDUCING MATTERS, TOTAL NITROGEN AND ALBUMINOIDS, STARCH, CANE SUGAR AND MILK SUGAR, CACAO MATTER.

IN this chapter, the analyses of the ingredients, added to cacao in the preparation of chocolate and, sometimes, themselves present in the nibs to a small extent, will be described.

Apart from the possible additions of cacao butter and alkali which have already been dealt with, a plain chocolate will contain added cane sugar, of which cacao contains only a small amount, naturally present. Starch may also be found to be present in cocoa and chocolate powders and eating-chocolates, though, in all cases, it should be looked upon as an adulterant, unless added for a specific purpose and duly notified on the package.

In milk chocolates, the addition of the solids of milk will increase the reducing matters, due to the presence of milk sugar or lactose, as well as the nitrogenous matters, due to the casein which milk contains. By the addition of the milk solids of full-cream milk, a new fat, butter fat, is also introduced, which further complicates analysis.

The direct estimation of cacao matter, present in a preparation, is not possible by chemical means, and, as this value is of great importance, owing to the regulations and standards of purity existing in many countries and to the fact that a rebate may be obtained on the amount of cacao matter exported, which has paid duty on entering this country, both the manufacturer and chemist should be in a position to state how much cacao matter any preparation contains.

To this end, the characteristic qualities of cacao mass and of its components have been observed, so that an estimation of one, or several, of the components, by direct or indirect means, may be converted into terms of cacao matter present.

### Reducing Matters.

The reducing-power of a substance is the extent to which that substance is capable of reducing a standard solution of copper, with the precipitation of cuprous oxide.

Certain sugars, such as glucose, lactose, etc., possess reducing-power, and, if the conditions of strength of solutions, temperature and time are kept constant throughout, a given weight of the sugar will precipitate a uniform quantity of cuprous oxide (estimated as cupric oxide), constant for that sugar.

The average reducing-power of 100 grammes of dry fat-free cacao is about five parts of cupric oxide. The addition, therefore, of even small quantities of soluble reducing matters will greatly increase the reducing-power of the cold-water extract of the cocoa preparation.

The reducing matters of pure cacao will include the small quantities of glucose, etc., which, as has been shown in former chapters, are present in the original beans.

#### *Method I. (by Fehling's Solution).*

The standard solution of copper is made up as follows:—34.5 grammes of copper sulphate are dissolved in 450 ccs. of water, and the solution accurately made up to 500 ccs.

173 grammes of Rochelle salt (sodium potassium tartrate) and 63 grammes of anhydrous sodium hydrate are dissolved in water, and the solution made up to 500 ccs.

The two solutions should be kept separate, but, for use, equal volumes of the two solutions are taken.

A very slight reduction takes place on heating the two

solutions (about 3 mgrms. of cupric oxide for every 50 ccs. of the mixed solutions), and this should be estimated, if great accuracy is required.

The reducing matter, to be measured, should be so regulated that from 150 to 350 mgrms. of cupric oxide are precipitated.

Fifty ccs. of the mixed Fehling's solution are placed in a wide-mouthed flask, of 250 ccs. capacity, and immersed in boiling water, until the temperature of the solution is stationary. An accurately weighed or measured solution of the reducing matters is then added, and the whole made up to 100 ccs. with boiling, distilled water. The heating is continued for exactly twelve minutes, when the solution is filtered through a double filter-paper, and the residue of cuprous oxide thoroughly washed and dried. The cuprous oxide is converted into cupric oxide by slowly charring the filter-papers and contents and by heating the residue in a weighed porcelain dish, first to a dull red, then to a bright red heat in a muffle furnace or over a Bunsen flame. The cupric oxide may then be weighed direct, converted into copper by reducing with hydrogen, or, after dissolving the oxide in nitric acid, may be deposited electrolytically as copper.

*Method II. (by Pavy's Solution).*

The copper solution is prepared by dissolving 20.4 grammes of Rochelle salt and 20.4 grammes of caustic potash in 200 ccs. of water. In another 200 ccs. of water, 4.158 grammes of copper sulphate are dissolved. The two solutions are then mixed, and, when cold, 300 ccs. of strong ammonia (S.G. 0.880) are added, and the whole made up to 1,000 ccs. with water.

The solution is standardised against a known weight of inverted cane sugar, when it will be found that 10 ccs. of Pavy's solution, prepared as above, diluted to 20 ccs. with water, equal about 5 mgrms. of glucose.

Estimations with this solution are made as follows:— Ten ccs. of Pavy's solution are placed in a wide-mouthed flask, and an equal bulk of water is added. A rubber stopper with two holes is fitted into the flask; into one hole is tightly placed the tapering end of a burette, whilst into the other is fitted a long glass tube, preferably extending to the open air, to carry away the ammonia fumes.

The burette is now filled with the solution containing the reducing matter, and the flask containing Pavy's solution brought to the boiling-point. The stopcock of the burette is opened, and a small quantity of the solution is run into the flask, and the whole brought again to the boil. Further additions of the solution are run in, and, after each addition, the flask and contents are boiled up. The blue colour of Pavy's solution will be seen to fade, after each addition and boiling, and the estimation is finished when the blue colour has completely faded.

Ten ccs. of Pavy's solution being equivalent to 5 mgrms. of glucose (by experiment), the reducing-power of the solution to be determined, in terms of glucose, is readily calculated from the number of cubic centimetres of that solution required to decolourise 10 ccs. of Pavy's solution.

#### Total Nitrogen, and Albuminoid or Proteid Matter.

In unadulterated plain cocoa and chocolate, the cacao matter is the only nitrogen-containing constituent. In milk chocolates, however, the presence of casein, in the milk solids employed, will increase the nitrogen and, consequently, the albuminoid value.

The use of ammonia in the production of "soluble" cocoa powder may also increase the amount of nitrogen, found by the Kjeldahl method, recommended.\*

\* A. Stutzer, *Zeitsch. angew. Chem.*, 1892, 510.

TOTAL NITROGEN (KJELDAHL'S METHOD, MODIFIED BY  
GUNNING, ARNOLD, ETC.).

One gramme of the sample is introduced into a flask of hard Jena glass and treated with 20 ccs. of strong sulphuric acid, a small globule of mercury being added at the same time. The flask is loosely closed by a ball stopper and gradually heated over a naked Bunsen flame. After the frothing has ceased, the heat is increased till the whole is briskly boiling, when 10 grammes of purest potassium bisulphate (absolutely free from any ammonium salts) are added. The flask and contents are heated, until the solution is quite clear and colourless, when they are cooled down, and the latter washed into a large distilling-flask bearing a doubly perforated cork. Through one hole of the cork is passed a thistle funnel, provided with a stopcock, and through the other a tube, connected to a condensing-apparatus.

A sufficient quantity of a solution of sodium hydrate (containing a little sodium sulphide) is passed through the funnel, just more than is necessary to neutralise the acid solution in the flask, and some small pieces of zinc foil are added, to prevent bumping, when the contents of the flask are boiled.

The ammonia is distilled off and collected at the end of the condenser into a known volume of  $\frac{N}{10}$  sulphuric acid, the loss of acidity of the standard acid being later estimated by titration with  $\frac{N}{10}$  sodium carbonate or hydrate solution.

The loss of acidity of the standard acid solution is due to the absorption of ammonia, distilled from the alkaline solution in the flask, and, when this value is known, the equivalent of nitrogen in the ammonia, originally present in the sample, can be readily calculated.

The total nitrogen multiplied by 6.25 will give, approximately, the albuminoid matter.

If, however, it is required to estimate the amount of nitrogen from the vegetable albumen alone, it is necessary to prepare the sample before submitting it to the Kjeldahl process.

#### ESTIMATION OF NITROGEN FROM ALBUMEN (STUTZER'S METHOD).

From 1 to 2 grammes of the sample are finely divided and boiled for a few minutes with 100 ccs. of water: 0.4 gramme of moist copper hydrate is added, and, after cooling, the liquid is filtered through a filter-paper, and the residue thoroughly washed with water. The filter-paper and contents are then submitted to the Kjeldahl process.

The difference between the total nitrogen and that found by Stutzer's method will give the non-albuminoid nitrogen, which, in the case of pure cacao, will result, almost entirely, from theobromine and caffeine.

In all cases of estimation of nitrogen by Kjeldahl's method, a blank experiment should be conducted side by side with the actual determination, in order to eliminate the errors, caused by any impure and nitrogen-containing chemicals.

#### Starch.

The amount of starch, present in pure cacao, never exceeds 12 per cent., the usual quantity, found, being 9 per cent. The addition of foreign starch, which, as a cheap adulterant, may be found in the commonest chocolate and in cocoa powders and, also, in the majority of chocolate powders, will rapidly increase the starch figure.

Bordas has shown that the percentage of starch, found in commercial cacao, is practically a constant, amounting to 9.34 per cent. in cacao mass and 28.5 per cent. calculated on cacao matter insoluble in water. On this assumption, he calculates the approximate amount of cacao matter present in a preparation and is able to estimate the extent



of adulteration with foreign starch, if such has been detected under the microscope. The starch may be estimated by any of the recognised methods, by hydrolysis of the starch by diastase or mineral acids, and estimating the sugars formed.

The results, obtained by Winton\* and others, show 23·66 per cent. of crude starch in cacao nibs, and 12·59 per cent. in the shell. It has been indicated, however, that the percentage of starch in the shell is quite small, and, for this reason, the results of many experimenters are in error. Revis and Burnett† have, more recently, criticised the various methods, employed, and, from careful comparison, have arrived at the conclusion that cacao shell contains no true starch. Bolton and Revis have found 19 to 23 per cent. of starch in the nib, and 9 to 10 per cent. in the shell, by employing a modified form of Ewer's method, but the former workers are certain, as we ourselves are, that the work of Davis and Daish‡ has provided us with the most accurate method of working, and they have, accordingly, estimated the starch in nibs and husk by these means. The actual method, employed, was :—

#### Method I.

Five grammes of fat-free dry cocoa matter are weighed out into a beaker, thoroughly stirred with 50 ccs. of 10 per cent. alcohol (by volume) and filtered under pressure—for preference in a large Büchner funnel, as the process is thereby considerably hastened. The cocoa is then washed with two further quantities of 50 ccs. of 10 per cent. alcohol and, finally, with 10 ccs. of 95 per cent. alcohol (by volume), taking care that, at no stage, the cocoa sucks dry, as it is then more difficult both to wash and to remove from the filter. The alcohol washing is very important, as other-

\* Winton, Silverman and Bailey, *Ann. Rep. Conn. Agric. Expt. Stn.*, 1902, 270.

† C. Revis and H. R. Burnett, *The Analyst*, 1915, xl., 429.

‡ Davis and Daish, *Jour. Agric. Soc.*, 1913, v., 437; *The Analyst*, 1913, xxxviii., 504.

wise great difficulties occur later on, and erroneous results are obtained. The moist cocoa is then scraped and washed out of the funnel into a 250 cc. flask with about 125 ccs. of boiling water, well mixed, and the flask placed in boiling water, with constant shaking, for 15 minutes to gelatinise the starch. The contents of the flask are then cooled to 38° C., mixed with 0.05 gramme of taka-diasase (obtainable from Messrs. Parke, Davis & Co.), rubbed up with a little water, 2 ccs. of toluene added, and the flask, after shaking and stoppering, placed in an incubator at 38° C. for 24 to 36 hours, shaking at intervals. At the end of this period, 10 ccs. of  $\frac{N}{10}$  NaOH are added to stop the action, the flask is cooled to 15° C., about 100 ccs. of water added, then 10 ccs. of acid mercuric nitrate solution run in on the surface, and, finally, water to the mark below the toluene. The contents of the flask are then mixed and filtered. To 100 ccs. of the filtrate (which should be perfectly colourless and bright), carefully measured into a flask, are added 0.5 gramme of sodium phosphate ( $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ ) and, after this has dissolved, 10 ccs. of sodium hydroxide solution are run in, the solution being agitated as this is done. The contents of the flask are mixed and filtered, and, in the case of 5 grammes of cacao matter, 50 ccs. are used for the gravimetric copper determination, the polarimetric readings being also made on this filtrate.

The solution of sodium hydroxide, used above, must be so adjusted that 10 ccs. just neutralise 4 ccs. of acid mercuric nitrate solution and should on no account be in excess, it being preferable to leave the solution slightly acid after addition of the alkali. In the experiments under consideration, the solution contained 55 grammes of ground caustic soda (97 per cent. NaOH) per litre. It was carefully determined that this treatment with acid mercuric nitrate caused no loss of starch conversion products.

The acid mercuric nitrate solution was made as follows :

Pure mercuric oxide (not mercury, as recommended by Wiley) was dissolved in twice its weight of nitric acid (S.G. 1.42), and the resulting solution diluted to 5 times its volume with water.

The results obtained by Revis and Burnett on cacao shell and nibs are shown as follow :—

Sample of shell.	CuO weighed.	Ventzke degrees.
(1) Blend . . .	0.038 grm.	+ 0.04
(2) Guayaquil . . .	0.031 „	+ 0.01
(3) Trinidad . . .	0.022 „	+ 0.03
(4) Grenada . . .	0.024 „	+ 0.05

In these cases, the copper oxide was that obtained from 50 ccs. of the final solution, used, and the polarimeter figures were the actual Ventzke saccharimeter degrees, obtained on polarising the filtrate after the acid mercuric nitrate and phosphate treatment, and they have not been calculated to the original shell.

Cacao nibs.	Per cent. of starch in fat-free nibs
Accra (low-grade cacao) . . .	12.8
Guayaquil . . .	8.0
Trinidad . . .	14.5
Grenada . . .	12.3
San Thomé . . .	12.4

*Method II. (Dubois\* Method for Cocoa Preparations in the Absence of Sugar).*

Two grammes of the sample are transferred to a 500 cc. Erlenmeyer flask, to which are added 20 ccs. of water and 12 ccs. of strong sulphuric acid. The latter is cautiously added with a slow rotation of the flask. The mixture is heated over a low flame with constant rotation, until the

\* W. L. Dubois, U.S. Dept. of Agric., 1910, Bull. 132.

colour changes from brown to reddish black. The time required for this change has been found to be approximately one and one-fourth minutes, so that all samples should be treated for this time.

Thirty ccs. of water are then added, the mixture heated to boiling and boiled for 15 seconds. A little cold water is poured in, the flask quickly cooled, and the acid nearly neutralised with a saturated solution of caustic potash.

The solution is then again cooled and transferred to a 250 cc. flask, completing the volume with cold water; 50 ccs. of the filtrate are used for the determination of copper-reducing substance as dextrose (100 mgrms. dextrose = 0.2538 gramme copper oxide = 0.2027 gramme of copper).\*

*Method III. (for Cocoa Preparations containing Sugar and Soluble Carbohydrates).* • • •

Four grammes of the sample are shaken with petroleum ether, until the whole of the fat has been removed.

After filtration and a further washing with ether, 100 ccs. of water are added to the contents of the flask, and the residue on the filter-paper is washed back into the flask. The flask is thoroughly shaken, and the contents filtered through a filter-paper. After three or four washings with water, and after the whole of the sugar has been removed, the filtrate is made up to 500 ccs. and may be used for sugar estimation, whilst the residue is washed into an Erlenmeyer flask and treated as described in *Method I.* for estimation of the starch.

Dubois, by employing these methods, obtained 10.77 per cent. to 13.05 per cent. of starch in cacao nibs, 11.38 per cent. to 13.64 per cent. in bitter chocolate, and 7.4 per cent. to 8.5 per cent. in sweet chocolate. In later experiments, the same author obtained 16.3 per cent. to 19.8 per cent. of starch in cocoas, and 10.4 per cent. to 18.2 per cent. in bitter chocolates.

\* Brown, Morris and Millar, *Jour. Chem. Soc. (Trans.)*, lxxi., 281.

Welmans\* has estimated the quantity of commercial dextrin added to chocolate: The dextrin is detected by treating the cocoa preparation, after extraction of fat, with water. The solution is filtered, and, to 10 ccs. of the filtrate, 40 ccs. of 90 per cent. alcohol added, when, if dextrin is present, an immediate turbidity is produced. The dextrin may be quantitatively precipitated by lead acetate in the presence of ammonia.

#### MICROSCOPICAL ESTIMATION OF STARCH.

Revis and Burnett have stated that starch cannot be detected in cacao shell under the microscope, and we admit to a difficulty in identifying it. In cacao nibs, the starch can be easily seen after staining with iodine solution. In cocoa powders, etc., added starch can, of course, be readily identified under the microscope by the same means, and, though, doubtless, with practice, an approximate estimation of the quantity, present, could be made, the microscopical method, suggested by Purvis and Hodgson, is not entirely satisfactory, as we have demonstrated elsewhere, when working with starchy products. Nor do we agree entirely with Purvis and Hodgson in their general statement, which runs as follows:—

“The presence of added starch in cocoa is easily detected by a microscopical examination; but its exact estimation by chemical methods is almost impossible, owing to the presence in cocoa of natural starch in varying quantities; the simplest method for determination is a microscopical one, and the results obtained are as near the true amount as those obtained by the most exact chemical methods.”

“Standard mixtures of cocoa and the starch shown under the microscope are made (5, 10, 15, 20 per cent. etc., of

\* P. Welmans, *Zeitsch. öffent. Chem.*, v., 478.

starch), and these are compared under the microscope with slides of the sample; with a little practice the standard containing the same percentage of starch as the sample can easily be picked out."

### **Sugar (Sucrose or Cane Sugar, Lactose or Milk Sugar).**

When solutions of cane sugar are boiled with mineral acids, the sugar is split up, or inverted, into two glucoses, the one rotating a plane of polarised light to the right and hence called dextrose, the other rotating to the left and called lævulose. These two sugars are capable of reducing copper solutions, as already described.

Carbohydrates, among which cane sugar, milk sugar, etc., are numbered as being soluble in water, are usually estimated by a combination of physical and chemical processes. When the sugars are present individually, their determination is not a matter of great difficulty, but, when more than one sugar is present in a preparation, their isolation and determination may be one of considerable complication.

In the case of cacao mass, the reducing matters, present, have already been shown to be small, and the power, possessed by a cold-water extract of cacao, of rotating polarised light is negligible. If, therefore, as is usually the case with a plain chocolate or chocolate powder, the sugar present is cane sugar only, the direct estimation of the sugar becomes a simple process, by testing the reducing-power of an inverted cold-water extract of a weighed sample, or by testing its power of rotating polarised light.

In this chapter, the process of testing the reducing-power of a cold-water extract has been described under "Reducing Matters," though, in order to render cane sugar capable of reducing a copper solution (either Fehling's or Pavy's), it is necessary to invert it.

*Method I.**Inversion of Cane Sugar, and Estimation by Reduction  
of Copper Solutions.*

To the clarified aqueous solution, containing the sugar from a weighed quantity of chocolate, add a few cubic centimetres of strong hydrochloric acid, and heat to boiling for ten minutes. Then place the vessel, containing the solution, aside for about ten hours, and estimate the inverted sugar by Pavy's or Fehling's solution (*vide* "Estimation of Reducing Matters," *Methods I. and II.*).

If the copper solutions have been previously standardised against a solution of inverted, pure cane sugar of known strength, the value of a given number of cubic centimetres of copper solution for complete precipitation or decolorisation, in terms of cane sugar, will be known.

*Method II.**Estimation of Cane Sugar by Polarimetric Methods.*

The power of rotating polarised light, possessed by the sugars, provides a ready method for their estimation.

The apparatus, in which such estimations are made, consists, essentially, of one stationary Nicol prism and a rotating prism attached to a circular scale. According to the instrument used, the scale is made to give readings as degrees or as direct percentage of cane sugar (saccharimeter). The first prism polarises the light, whilst the second determines the plane of the polarised light coming from the first.

The means of obtaining the readings are too well known to need description here.\*

The estimation of a sugar in solution can be made (if only one sugar is present) by observing the angle of rotation of that solution in a tube of known length, when, if the specific

\* For full particulars, see Landolt, "Optical Rotation of Organic Substances," 1902, 325.

rotatory power\* of the particular sugar is known, the following equation will serve for determining the weight of sugar in 1 cc. of the solution:—

$$P = \frac{a}{[a]l} \text{ where } a = \text{observed rotation of the solution.}$$

- $[a]$  = specific rotatory power of the particular sugar,
- $l$  = length of observation-tube in decimetres.

As an example: The observed rotation of a cane sugar solution in a 1 decimetre tube is  $5^\circ$ , and, the specific rotatory power of cane sugar being  $+ 66.5^\circ$ , the percentage content of sugar in that solution

$$= \frac{5 \times 100}{66.5 \times 1} = 7.518.$$

The specific rotatory powers of individual sugars have been carefully estimated by a number of observers; thus, sucrose (cane sugar) =  $+ 66.5^\circ$ ; maltose (malt sugar) =  $+ 138.0^\circ$ ; lactose (milk sugar), anhydrous =  $+ 55.4^\circ$ ; lactose, crystalline =  $+ 52.6^\circ$ ; dextrose =  $+ 51.3^\circ$ ; lævulose =  $- 95.4^\circ$ ; invert sugar =  $- 22.0^\circ$ .

In saccharimeters, or polarising apparatus especially adapted for determining the quantity of cane sugar in solutions, the scale, attached to the analysing prism, is divided so as to read the percentage of cane sugar direct.

The official method for determining the percentage of cane sugar in chocolate, according to the German decree of May 31st, 1891, is as follows: 13.024 grammes of chocolate are damped with alcohol, then warmed for fifteen minutes with 30 ccs. of water on a water-bath. Whilst still hot, the solution is poured on to a wet filter-paper, and the residue again treated with hot water, until the filtrate nearly equals 100 ccs. The filtrate is mixed with 5 ccs. of basic lead acetate solution, allowed to stand for fifteen minutes,

\* The specific rotatory power of a sugar is the number of degrees of rotation recorded, when 1 gramme of the sugar is dissolved in 1 cc. of fluid and observed, by yellow light, through a tube 1 decimetre long.



clarified with alum to which a little alumina has been added, made up to a definite volume (110 ccs.) and polarised.

In plain chocolates and chocolate powders, it is improbable that any sugar other than cane sugar will be found, though glucose was used during the war. In special preparations, inaltose, lactose, etc., will occur, due to the addition of malt powders, milk solids, etc., and, consequently, variations in the methods of estimation will be necessary.

As milk chocolates have now become so popular, it is intended to give methods by which lactose can be determined in the presence of cane sugar. The following method, which has been devised by Dubois,\* was adopted as a provisional method by the A.O.A.C.

### *Method III.*

#### *Estimation of Lactose and Sucrose in Milk Chocolate Preparations (Dubois' Method).*

Thirteen grammes of the sample are freed from fat by shaking and centrifuging with two separate quantities of gasoline (100 ccs.). The solvent is separated by decantation, and the residue treated with 100 ccs. of water and shaken for ten minutes.

Five ccs. of basic lead acetate solution (for preparation, see later) are added, the precipitate filtered off, and the excess of lead removed from the filtrate (original filtrate).

Twenty-five ccs. of this solution are allowed to stand overnight, to destroy bi-rotation, and then polarised. Multiply the readings, so obtained, by two (*Result A.*).

Invert (for method of inversion, see later) 50 ccs. of the original filtrate, and nearly neutralise the acid, after cooling, with sodium hydrate solution. Make up the combined solution to 100 ccs. with water, and bring to the temperature

\* W. L. Dubois, *U.S. Dept. of Agric., 1907, Bull. 107.*

at which the direct readings were taken, and polarise. Multiply the readings by 4 (*Result B*). Using the same solution, polarise again at 86° C. in a water-jacketed tube, and multiply the readings by 4 (*Result C*).

The approximate weights of sucrose and lactose, present in the 18 grammes of sample, are obtained by the following formulæ:—

$$\begin{aligned}\text{Grammes of sucrose} &= \frac{(A - B) \times 1.05 \times 13}{142.66 - \frac{t}{2}} \\ \text{Grammes of lactose} &= \frac{C \times 1.264 \times 1.11 \times 1.05 \times 13}{100} \\ &= \frac{19.152 C}{100}\end{aligned}$$

In these formulæ, the factors are explained as follow \* :—

1.05 is the factor to allow for the increase in volume of the solution occurring on addition of 5 ccs. of lead acetate solution.

1.264 is the factor allowing for the difference in normal weight of sucrose and lactose, thus  $\frac{32.884}{26.00} = 1.264$ .

1.11 is the factor for reducing readings taken at 86° C. to readings taken at 20° C.

*A* = Direct readings of normal weight of material.

*B* = Invert readings of normal weight of material.

*C* = Invert readings of normal weight of material at 86° C.

From the total amount of sugar, found by this method, the percentage of sugars, present, is calculated by the following formulæ:—

$$\begin{aligned}\frac{(A - B) 1.05 X}{142.66 - \frac{t}{2}} &= \text{Percentage of sucrose.} \\ (1.472) X &= \text{Percentage of lactose.}\end{aligned}$$

This correction is necessary from Dubois' experiments, conducted at 20° C., to the following extent :—

For 2 grms. of sugar in sample	X = 101.20
„ 4 grms. „ „	„ X = 102.50
„ 6 grms. „ „	„ X = 103.60
„ 8 grms. „ „	„ X = 104.80
„ 10 grms. „ „	„ X = 106.05
„ 15 grms. „ „	„ X = 109.40
„ 20 grms. „ „	„ X = 112.40

*Provisional Official Method of Inversion.*

To 5 ccs. of the clarified solution, free from lead, add 5 ccs. of 38.8 per cent. hydrochloric acid solution, and set aside for twenty-four hours at a temperature not below 20° C., or, if the temperature be above 25° C., for ten hours.

*Preparation of Lead Acetate Solution.*

Boil 430 grammes of normal lead acetate, 13 grammes of lead oxide (PbO) with 1,000 ccs. of water for half-an-hour. Cool the mixture, and allow to settle. Decant the clear solution, and dilute with recently boiled water till the specific gravity = 1.25.

This provisional method has been under survey by a number of workers, and it must be recorded that the results obtained are far from satisfactory. The form in which the method is now before the American Association for consideration, prior to adopting it as a standard method, is materially different from that given in the foregoing pages. The determination of lactose by polarising at 86° C. is discontinued, the calculation of this constituent being made entirely from the same polarisations as are used in the sucrose calculations.

## Method IV.

*French Official Method for estimating Lactose in  
presence of Sucrose.*

A very close approximation to the correct lactose value in milk chocolates can be made by estimating, direct, the copper reducing power of a standard solution. This, in effect, is the official French method (*Ann. des Falsif.*, iv., 417) which, in the case of milk chocolate, recommends taking the residue from the fat estimation (3 grammes of original chocolate) and treating it with 30 ccs. of a 1 per cent. solution of phosphorus tri-iodide for half-an-hour, at a temperature of 60° C. The mixture is agitated, separated in a centrifugal, and the clear liquor poured off. This treatment is to be repeated three times, and the combined solutions made up to 150 ccs. Lactose is estimated, direct, on a portion of the solution with Fehling's solution. Sucrose is inverted and similarly determined. In the absence of invert sugar, or other reducing sugars besides lactose, this method is to be recommended, as we have shown in a number of analyses of milk chocolates of known composition.

The estimation of lactose, in the presence of sucrose and invert sugar, has been the subject of much study, of which only a brief survey can be given here. The necessity for the determination has arisen chiefly for the purpose of obtaining complete analyses of sweetened condensed milk, infant foods, etc.

In 1906, the A.O.A.C. considered the question of obtaining results by (a) gravimetric and volumetric methods with Fehling's solution, (b) polarimeter method, before and after inversion with citric acid and yeast, and, in the case of unsweetened milk, by direct gravimetric and polarimeter methods. The results, given on p. 524, were obtained.

	Gravimetric method.		Polariscopic method.	
	Lactose.	Sucrose.	Lactose.	Sucrose.
	Per cent.	Per cent.	Per cent.	Per cent.
<b>SAMPLE A.</b>				
<i>Sweetened condensed milk:</i>				
Fulton (Mass.)	13.37	40.62	—	—
Norton (Arkansas)	9.5	45.48	—	—
Bartlett (Maine)	14.94	40.18	—	—
Jaffa and Stewart (California)	14.37	41.78	10.88	40.00
Olson (Wisconsin)	14.69	35.25	—	38.67
<b>SAMPLE B.</b>				
<i>Unsweetened condensed milk:</i>				
Fulton	10.49	—	—	—
Norton	9.4	—	11.3	—
Bartlett	10.08	—	—	—
Olson	11.05	—	11.56	—
Patrick and Boyle (Washington)	11.04	—	10.07	—

Other determinations were made on one sample of sweetened condensed milk and four samples of unsweetened milk.

	Gravimetric method.		Polariscopic method.	
	Lactose.	Sucrose.	Lactose.	Sucrose.
	Per cent.	Per cent.	Per cent.	Per cent.
<i>Sweetened condensed milk:</i>				
Olson (Wisconsin)	16.41	35.91	—	34.35
<i>Unsweetened condensed milk:</i>				
Jaffa and Stewart (California)	9.28	—	9.36	—
Olson	9.91	—	9.19	—
	8.11	—	8.00	—
Jaffa and Stewart	10.87	—	10.74	—
	9.96	—	9.84	—

Baker and Hulton,\* in 1910, estimated lactose in the presence of the commonly occurring sugars, and first attempted Tollens' method by the use of masic acid. This method was abandoned as unsatisfactory and as quite useless when working with small quantities of lactose in the presence of other organic matters. Eventually, they evolved a method with use of brewer's yeast, experimental data for

\* J. L. Baker and H. F. Hulton, *The Analyst*, 1910, xxxv., 512.

which can be found in the original papers. These workers obtained the following results :-

Materials used.	Grammes of lactose present in solution before fermentation.	Grammes of lactose per 100 ccs. of solution titrated.		Per cent. Lactose determined.
		Calculated.	Found.	
Lactose alone	10	8	7.6	95
" "	6	6	5.44	90.5
" "	3	0.3	0.27	90.0
Lactose + trace of glucose	2	1	0.5	95.0
" + glucose (equal weights)	1	0.8	0.74	92.0
" + " " "	3	2.4	2.37	98.7
" + invert	2	1.5	1.45	98.5
" + glucose + maltose (equal weights)	2	2	2.01	100
" + " "	1	1	0.96	96
" + cane sugar + maltose (equal weights)	2	2	2.05	102
Wheat starch containing 10 per cent. lactose	2	0.8	0.8	100
Flour containing 5 per cent. lactose	1	0.4	0.395	98.5
" " 10 " "	0.5	0.33	0.314	94
" " 10 " "	0.5	0.33	0.32	97
" containing 10 per cent. lactose + 10 per cent. cane sugar	0.5	0.33	0.32	97

In 1911, some official French methods for the estimation of lactose were published in the *Annales des Falsifications* (iv., 420), and we have already considered some of the results obtained.

•The year 1914 saw several papers published on the subject, among them being those by Revis and Payne,\* and Jitendra Nath Rakshit.† In the latter paper, an excellent

Percentage of lactose.		Percentage of sucrose.	
Found.	Calculated.	Found.	Calculated.
4.80	4.41	4.73	4.76
4.24	4.32	9.03	9.09
3.66	3.66	16.60	16.67
3.12	3.09	33.40	33.33

C. Revis and G. A. Payne, *The Analyst*, 1914, xxxix., 476.

Jitendra Nath Rakshit, *Jour. Ind. Eng. Chem.*, 1914, vi., 207.

*résumé* of the existing knowledge of lactose determination is given, and a modified "French official" method devised. Working with milk of known lactose value, various quantities of cane sugar were added, and the two sugars were analysed by the method proposed, with the results shown above.

The author then turned to some commercial brands of sweetened condensed milk, estimated the sugars by that method, and determined the ash, proteid matter, etc., comparing the added results with the total solids, obtained by evaporation.

The following table shows the results:—

Brands	Ash proteid, fat, lactose, sucrose.	Total solids by evaporation.
1. Best skimmed condensed milk	70.7	70.2
2. Nestlé's condensed milk, Nest brand	74.2	74.8
3. Milkmaid brand	70.8	70.1
4. Best skimmed condensed milk, Cow and Calf brand	75.1	75.6

In 1918, Grossfeld\* provided empirical formulæ for calculating lactose in a mixture containing both cane sugar and invert sugar, the data, required, being the polarimeter value and the reducing-power of the mixture after inversion. The inversion was made under standard conditions, namely, 50 ccs. of the solution mixed with 2 ccs. of hydrochloric acid (S.G. 1.125), heated in a boiling water-bath for thirty minutes. The reducing-power is expressed in terms of invert sugar, and the lactose is then calculated from the formula.

$$L = 1.20 Z \frac{Dx - Di}{De - Di}$$

where  $Dx$  = Specific rotation found in invert sugar,

$De$  = Specific rotation of lactose (+70.6°),

$Di$  = Specific rotation of invert sugar (−16.7°),

$De - Di$  is thus +87.3° and the formula becomes:—

$$L = 0.01375 Z (Dx + 16.7).$$

\* J. Grossfeld, *Zeitsch. Nahr. Genussm.*, 1918, XXV., 249.

Sucrose is found from the formula—

$$S = 0.01145 Z (70.6 - D\%),$$

since 100 per cent. of cane sugar gives 100 per cent. of invert sugar, under the conditions given.

### Cacao Matter.

The amount of cacao matter, present in chocolate, etc., assumes a certain importance, seeing that a rebate is allowed on exported cacao matter which has previously paid duty on entering this country.

The direct estimation of cacao matter is, obviously, an impossibility, as cacao is composed of a number of complex chemical constituents, and the amount of cacao matter, actually present, can only be obtained by indirect means.

Undoubtedly, the most satisfactory method is complete analysis of the preparation, when, after it has been found that no foreign starch, mineral matter, husk, etc., have been added, the difference between 100 and the sum of the percentages of sugar (if any) and total fat will give a very close value for the amount of fat-free cacao matter present.

Booth, however, obtains the proportion of fat-free cacao matter on the assumption that the cold-water extract is, for all practical purposes, identical in husk and nib. The cold-water extract is assumed to be 12 per cent. of the total cacao, and, after the sample under investigation has been corrected for (a) the sugar, as ascertained by the polarimeter, and for (b) the extract of any cereal that may be present (7.7 per cent. for wheat, 5.1 per cent. for barley, 0.8 per cent. for maize or cornflour, 0.9 per cent. for rice, 1.98 per cent. for sago, and 0.4 per cent. for arrowroot), the residual figures multiplied by  $\frac{100}{24}$  will give the dry, fat-free cacao matter present.

If this agrees with that obtained by multiplying the corrected nitrogen by 20 (the factor for fat-free nib), then no shell is present. If it does not agree, then the following



formula is recommended for calculating the respective proportions :—

$$y = .40 N - x,$$

where  $x$  = the percentage of fat-free cacao matter, found as above,

$y$  = the percentage of fat-free nib,

$N$  = total nitrogen due to cacao matter.

In a husk-free cocoa containing 12 per cent. of cold-water extract, 50 per cent. of fat and 2.5 per cent. of nitrogen, the two equations will be found to agree, thus :—

$$\text{Dry, fat-free cacao matter} = \frac{12 \times 100}{24} = 50 \quad (1)$$

$$y = 100 - 50 = 50 \quad (2)$$

Bordas has made use of somewhat similar means for the estimation of the amount of cacao matter in a preparation ;

TABLE LXXXI.—Numerical Relations between the Various Constituents of Cacao.

	$\frac{S}{U-F}$	$\frac{F}{U-S}$	$\frac{S}{U}$	$\frac{F}{U}$	$\frac{S}{F}$
<i>Air-dried husked beans :</i>					
Maracaibo . . . . .	0.4289	0.7395	0.1636	0.6185	0.2645
Caracas . . . . .	0.4074	0.7273	0.1578	0.6125	0.2577
Trinidad . . . . .	0.3452	0.7297	0.1247	0.6387	0.1953
Guayaquil Machala . . . . .	0.3583	0.7330	0.1297	0.6379	0.2033
Porto Plata . . . . .	0.3660	0.7734	0.1481	0.6589	0.2247
Means . . . . .	0.3946	0.7406	0.1446	0.6335	0.2283
<i>Air-dried husks :</i>					
Maracaibo . . . . .	0.1390	0.0412	0.1341	0.0357	3.7564
Caracas . . . . .	0.1525	0.0356	0.1479	0.0304	4.8674
Trinidad . . . . .	0.1515	0.0467	0.1455	0.0399	3.6413
Guayaquil Machala . . . . .	0.1272	0.0402	0.1227	0.0352	3.4827
Porto Plata . . . . .	0.1850	0.0797	0.1728	0.0660	2.6202
Means . . . . .	0.1508	0.0484	0.1446	0.0414	3.4920
<i>Chocolate in cakes mixed with sugar only :</i>					
4.8 marks per lb. . . . .	0.3838	0.7363	0.1438	0.6253	0.2300
4.0 " " " " . . . . .	0.3476	0.7161	0.1314	0.6220	0.2112
3.2 " " " " . . . . .	0.3310	0.7226	0.1297	0.6354	0.1899
2.4 " " " " . . . . .	0.3029	0.7035	0.1141	0.6232	0.1831
2.0 " " " " . . . . .	0.3729	0.7490	0.1298	0.6517	0.1992
Means . . . . .	0.3480	0.7245	0.1282	0.6317	0.2029

thus, in a pure cocoa powder or preparation free from sugar, starch, etc., the following equations should hold good:—

$1.30 \times$  (cacao matter insoluble in water) = (dry fat-free cacao matter), and  $2.95 \times$  (cacao matter insoluble in water) = (dry cacao matter).

The factor 32.85, which is the value for "cacao matter insoluble in water," found to be practically constant for eleven different varieties of cacao, is applicable also to cocoas which have been rendered soluble by treatment with alkali.

In the estimation of cacao matter in a preparation, Bensemann's figures, which are given in Table LXXXI showing the relations between starch (S), fat (F) and the insoluble matter of cacao (U), may be found useful.

#### Estimation of Milk Solids in Milk Chocolate.

Estimations of nitrogen and nitrogenous matters, the chemical and physical constants of the fat and the estimation of the lactose, present, afford means by which the amount of total milk solids in the chocolate can be calculated. Seeing that milk solids from full-cream milk contain 25 per cent. of fat, 38.5 per cent. of lactose, and about 27.5 per cent. of nitrogenous matters, whilst cacao mass contains about 50 per cent. of fat (with different chemical and physical constants to butter fat), practically no reducing matters, and about 15.5 per cent. of nitrogen-containing bodies, the proportions of added milk solids can be readily estimated. By careful consideration of the chemical and physical constants of the fat, it can be determined whether the milk solids were obtained from full-cream milk, the Reichert-Meissl value being especially useful in determining the proportions of the fats present.

Those methods were reported upon by the U.S. Department of Agriculture,\* which provided figures showing

\* U.S. Dept. Agric., Bur. of Chem., 1914, Bull. 162.

satisfactory results obtained both in the determination of lactose and butter fat in milk chocolates of known composition. At the same time, a method for the estimation of casein was suggested, based upon Baier and Neumann's method of extracting the casein, from defatted chocolate, by sodium oxalate and precipitating with uranium acetate in acetic acid solution. The actual method, employed, with the results obtained by five chemists, are given below, and it should be remarked that the milk chocolates were obtained from a well-known manufacturer who submitted the formulæ from which the chocolates were made.

#### DETERMINATION OF CASEIN.

"Rub 10 grammes in a mortar, with the gradual addition of 1 per cent. of sodium oxalate solution, avoiding the formation of lumps, and pour into a 250 cc. flask, until 200 ccs. of the sodium oxalate solution have been used. Place the flask on an asbestos board, and heat slowly until the contents come to a boil, occupying at least 30 minutes in bringing it to this temperature. During the heating, have a funnel, the stem of which is closed by melting, rest in the neck of the flask. Fill to the mark with hot sodium oxalate solution, allow to stand, with frequent shaking, until the next day; fill to the mark with sodium oxalate solution at 20° C., mix, and filter through a folded filter. To 100 ccs. of the filtrate, add 5 ccs. of a 5 per cent. solution of uranium acetate, and then, drop by drop, with continuous stirring, add 35 drops (or approximately 1.5 ccs.) of 30 per cent. acetic acid. Separate the precipitate from the liquid by means of a centrifuge; wash three times with a solution containing 5 grammes of uranium acetate and 3 ccs. of 30 per cent. acetic acid in 100 ccs. This washing is quickly accomplished by shaking the precipitate in the centrifuge bottle with about 75 ccs. of the wash-solution and throwing out the solid material by centrifuge. Finally, wash the precipi-

tate on to a nitrogen-free filter with the washing solution, and determine the nitrogen in the precipitate, multiplying this result by 6.38 to obtain the casein.

"Five chemists, including the referee, tested the method as submitted, and in the table below, the results obtained are indicated. It will be noted that, in the chocolate containing most casein, the results are more accurate than in that sample containing less casein. The figures, however, are within a few tenths of a per cent. of the amount actually present in both cases, and it would seem that the method promises to be sufficiently accurate to be used in the judging of the quality of the class of products to which it is to be applied."

*Casein in Milk Chocolate.*

Analyst.	Casein.				Fat in original.		Casein originally present.	
	In fat-free substance.		Calculated to original.		No. 1. No. 2.		No. 1. No. 2.	
	No. 1.	No. 2.	No. 1.	No. 2.				
W. L. Dubois, Buffalo, N.Y.	5.50	4.84	3.68	3.24	32.61	33.55	3.91	2.82
E. Bloomberg, Galveston, Tex.	5.78	4.98	3.90	3.31	—	—	—	—
	5.79	4.92	3.90	3.27	—	—	—	—
H. E. Woodward	5.26*	4.63	3.54	3.08	—	—	—	—
	5.42	4.58	3.65	3.04	—	—	—	—
	5.59	5.32	3.77	3.54	—	—	—	—
L. B. Burnett, Washington, D.C.	5.43	4.84	3.66	3.22	—	—	—	—
	5.91	5.16	3.98	3.43	—	—	—	—
W. C. Taber, Washington, D.C.	5.15	4.21	3.47	2.80	—	—	—	—
	5.28	4.74	3.56	3.15	—	—	—	—

\* Low result probably due to solution boiling over.

DETERMINATION OF MILK FAT (BY REICHERT-MEISEL VALUE).

The associate referee of the U.S. Department of Agriculture, in considering this question, after a very great number of experiments, suggested the use of the following

formula for calculating the percentage of butter or milk fat present in the chocolate :—

Let  $A$  = grammes of butter fat,

$B$  = grammes of cocoa fat (cacao butter),

$C$  = Reichert-Meissl number of extracted fat,

0.5 = Reichert-Meissl number of cocoa fat (cacao butter),

24 = Reichert-Meissl factor for butter fat.

Then

$$\frac{24A}{5} + \frac{0.5B}{5} = C$$

$$A + B = 5$$

$$B = 5 - A.$$

Working out the formula, we get this value :—

$$A = \frac{C - 0.5}{4.7},$$

from which the following is deduced :—

$$\begin{aligned} \text{Per cent. butter fat in chocolate} &= \text{per cent. total fat} \\ &\times \frac{C - 0.5}{23.5} \end{aligned}$$

#### TOTAL MILK SOLIDS.

By applying the above methods and the polarimetric estimation of lactose, milk solids were determined in four chocolates by the chemists of the U.S. Department of Agriculture. The chocolates were of known composition, and the results of the analyses are shown in the table below. It should be observed that no allowance was made by these chemists for the ash in the milk solids, the total figures for which could, therefore, be increased by about 1 per cent.

*Milk Solids in Milk Chocolate.*

Sample.	Found by analysis.					Declared present.
	Casein.	Proteids. Casein × 1.25.	Lactose.	Butter fat.	Total.	
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
B . . .	3.39	4.00	6.56	4.79	15.58	16.25
1 . . .	3.68	4.60	6.58	6.36	17.54	17.24
2 . . .	3.24	4.05	4.89	3.23	12.17	12.41

## CHAPTER XXXII

### ADDED HUSK—ITS DETECTION IN COCOA PREPARATIONS

FROM the law cases of the last few years, dealing with the question "What is Chocolate?," we may readily believe that, provided a confection contains an amount of cacao matter sufficient, in the magistrate's opinion, to justify the title—that is *chocolate* in this country. It is all very unsatisfactory; the magistrate has no standard to which to work, and the solicitors may put forward any nonsense without contradiction. In 1909, there were two cases which might have had some influence upon the standardisation of cocoa products, but we read that the certificate of analyses, which showed the addition of foreign fat, did not distinguish between coconut fat and cacao butter, and, again, that a statement was made that "Messrs. Suchard, the well known makers, tell you that chocolate is made simply and solely from the cocoa bean, and there may be added a small proportion of sugar and preservatives—that is chocolate."

The first case was a summons in respect of a sample of plain chocolate drops, the analysis of which showed 18·5 per cent. of foreign starch, 22·9 per cent. of foreign fat, and that the fat-free cacao matter contained not less than 27 per cent. of its weight of cacao shell or husk. The second summons was in respect of a sample of broken milk chocolate, the analysis of which showed 12 per cent. of foreign starch, 19·25 per cent. of foreign fat, and that the fat-free cacao matter contained not less than 18·5 per cent. of cacao shell or husk. The magistrate imposed a fine of 20s. and costs

in respect of the first summons, and 2s. and costs in the second, after much talk between the solicitors as to whether a confection containing a percentage of cacao matter (husk or no husk) amounting to 10 or 11 was sufficient to justify the appellation of "chocolate."

Another case occurred in 1910, when a grocer was summoned for "selling a cocoa powder containing 60 per cent. of powdered cocoa shells." The magistrate admitted that he had to decide "What is Cocoa?" and adjourned the case, in order to have another portion of the powder analysed by the Government analyst and to enable himself to look up the authorities on the matter.

Apart from the difficulties that must confront the magistrate, the difficulties encountered by the analyst are equally troublesome. It is quite an easy matter to determine 60 per cent. of cacao shell in a cocoa powder, but it is less easy to estimate small quantities, unless a definite method of analysis be fixed. The author well remembers approaching the customs analysts with regard to this question, with the idea that a satisfactory method might be established. The degree of suspicion with which the author was regarded and the secrecy maintained by these scientific authorities necessitated approach by another road.

Most chemists, dealing with foodstuffs, have been confronted with the problem at some time or other, and one must sympathise with such workers as Booth, Baker, Hulton and others, whose published efforts have always brought to light one fact, namely, that the results, obtained, depend upon the method of analysis, employed. For this reason, we are aware that the figures, given vaguely as fibre, crude fibre, woody fibre, cellulose, etc., in tables of analyses in foregoing chapters of this work, are of little, if any, use to the casual reader, unless the method of analysis be given, but we are also aware that, with a little intelligence, these figures are valuable when compared one against the other,

provided that they have been obtained by the same experimenter.

Further, so far as the chemist, dealing with cocoa and chocolate in a factory, is concerned, we know that reliable figures can be obtained by almost any of the recognised methods. Continuous practice in handling his raw materials and in analysing the finished products enables him to compare his results against standards which, of course, he must have set up. For the public analyst, the problem is less easy—the personal element is lacking and his knowledge of factory practice is insufficient—with the result that, unless a standard method is established, the estimation of husk in cocoa preparations becomes of little value.

In the excellent work of Baker and Hulton, to which we shall have occasion to refer more fully later, the authors state: "It is obviously necessary to use very careful judgment in the interpretation of the results by any one or all of the methods described. In some cases the agreement is good, in others the differences, as calculated from the levigation and nitrogen figures, are large. In estimating considerable proportions of shell, the analyst is on fairly safe ground, but when it comes to such small differences as separate Grades A. and B., the available methods are quite inadequate." We agree with this statement to a very large extent, but we would point out that no worker has, so far, taken the trouble to report analyses, if done at all, of selected mixtures from straight shell to straight cocoa powder, and to correlate the results. This is, of course, done in every cocoa and chocolate laboratory, using one method most reliable in the opinion of the analyst concerned.

In Chapter XXX, we have already referred to a number of methods recommended for the estimation of added husk to cocoa powders, which can be employed with success when intelligently used in the manner indicated above, but we propose to offer a few other methods, for examination by



those interested in the subject, and to test those, with which we are less familiar, ourselves, when time allows.

### Physical Methods.

The camps are divided into adherents of physical and of chemical methods, of the first of which the most important is the levigation method. We have already given the method for estimating fibre by the levigation process, as described by Baker and Hulton, based on a method of Macara, adapted from Filsinger. In 1910, Filsinger and Bötticher\* examined a method of flotation in a solution of calcium chloride of known strength, that had originally been suggested by Goske. Tests were made with commercial cocoa powders and roasted Arriba nibs, carefully picked free from shell, and on known mixtures of this with cacao shell. The results showed that separation was by no means complete, *e.g.*, an equal mixture of shell and nib yielded only 19.6 per cent. of shell by the Goske method and 53 per cent. by Filsinger's original elutriation method. The correction of subtracting 6 per cent., as the mean unavoidable shell-content, led, in the opinion of these experimenters, to quite impossible results, especially when dealing with very pure cocoa powders.

Ulrich,† in 1911, examined the then-existing processes of determination of shell in cocoa preparations. With regard to microscopical examination, this author remarks that the introduction of modern machinery, which is capable of reducing the cocoa to a very fine powder, has rendered this method very uncertain, and no accurate estimation of the proportion, present, is possible. Filsinger and Drawe's‡ elutriation method was then passed under examination and recorded as fairly accurate when the nib and shell had been ground together, as the resulting particles of shell were

\* F. Filsinger and W. Bötticher, *Zeit. öffent. Chem.*, 1910, xvi., 311.

† C. Ulrich, *Arch. Pharm.*, 1911, cclix., 524.

‡ F. Filsinger and Drawe, *Zeit. öffent. Chem.*, 1903, ix., 161.

considerably larger than those of the nibs, when reduced in this way. A factor, 1.72, has to be used, if the particles are of the same size, and good results are then recorded by this worker. The original factor, 1.48, by which the weight of the shell is multiplied to allow for that part of the shell which is soluble in water, was found to be insufficient, and its increase to 1.72 was suggested. Goske's method of flotation in calcium chloride solution was condemned, as by Filsinger, as useless. Ulrich finally suggested a method for estimating the fibre, present, based on the fact that "cacao-red" is present only in the nibs. This method we shall refer to later.

Goske's method again came under review by Schenk, Schmidt and Görbing,\* in 1912, and was again found to be unsatisfactory. The results, obtained by these three experimenters, showed that they were dependent upon the temperature at which the liquids were centrifuged and, also, on the speed of the rotation of the centrifuge. To show the effect of the degree of fineness of the material, it was stated that with very finely ground samples, containing 25 per cent. and 50 per cent. of shell, the values obtained by Goske's method were only 5 per cent. and 7.4 per cent., respectively.

Dubois and Lott,† in 1911, had already found Goske's method to be only approximately accurate, when the precise factor for the added shell was known, and quite unreliable for determination of husk in commercial cocoa powders.

The evidence against Goske's ‡ method for estimation of husk in cocoa powders by flotation in calcium chloride solution is generally so strong that it is unnecessary to give details of his method here. There is, however, a later water-modification of his process devised, described by Grosse§, which, conceivably, is an improvement. Grosse recommends

\* A. Schenk, F. Schmidt and J. Görbing, *Zeit. öffent. Chem.*, 1912, xi., 201.

† W. H. Dubois and C. I. Lott, *Jour. Ind. Eng. Chem.* 1911, iii., 251.

‡ A. Goske, *Zeitsch. Nähr. Genussm.*, 1910, xix., 154.

§ H. Grosse, *Chem. Zeit.*, 1915, xxxix., 816.

the following treatment: From 1 to 1.5 grammes of the cocoa are ground with 20 ccs. of hot water, boiled and centrifuged (about 500 r.p.m.). The liquid portion is decanted, the sediment mixed with 25 ccs. of hot water and again centrifuged. The sediment, now obtained, is found to be divided into two portions by a white layer, the upper portion being cacao mass, the lower and dark brown portion the cacao shell. The upper layers are removed, the lower layer twice centrifuged in a small tube, transferred to a basin, dried and weighed. The sediment should be ground and centrifuged again, if large pieces of cacao mass are found in the shell layer. The amount of shell, so obtained, is multiplied by three, to obtain the actual quantity present, as, by this treatment, cacao shell loses two-thirds of its weight. By this method, a cocoa powder, yielding a sediment corresponding to 6 per cent. of cacao shell, mixed with 33.3 per cent. of its weight of cacao shell, showed, on analysis, 31.6 per cent. of added shell. Doubtless, by systematic standardisation, this method could be modified to yield fairly accurate results, and it will be observed that it is very similar to that adopted by Baker and Hulton.

Kalusky\* found that the cell-tissue of the nibs, obtained by treating the fat-free substance with diastase and, subsequently, with hydrochloric acid, varied in specific gravity from 1.1131 to 1.3503 (usually 1.25), whilst that of the husk lay between 1.4324 and 1.9337 (usually 1.50). Separation was possible in a mixture of chloral hydrate, glycerol and water (S.G. at 17°C. = 1.415), and a mixture was eventually selected having a specific gravity of 1.5.

The method adopted by Kalusky is as follows:—One gramme of the dry, fat-free powder, (completely extracted with ether) is boiled for five minutes with 500 ccs. of water, then cooled to 65°C. and maintained at this temperature for one hour, after the addition of a small quantity of diastase.

\* L. Kalusky, *Zeitsch. Nahr. Genussm.*, 1912, xxiii., 654.

Twenty-five ccs. of 25 per cent. hydrochloric acid are added, and the mixture boiled for thirty minutes. The insoluble matter or tissue is filtered through a weighed filter-paper, washed with hot water, dried for two hours at 100° C. and weighed. The dry tissue of fibre is detached from the filter, ground finely in a mortar and again dried. A weighed portion is then mixed, in a suitable tube, with 20 ccs. of the medium (prepared as described below) and centrifuged for five minutes.

The medium is prepared by mixing 210 grammes of chloral hydrate, 50 grammes of glycerol and 35 grammes of water, and should have a specific gravity of 1.50 at 17° C. More chloral hydrate or water, as the case may be, is added to secure the desired specific gravity.

The portion of tissue, settled to the bottom of the tube, is separated, mixed with 100 ccs. of water, collected on a filter, washed with hot water, dried and weighed. The weight of heavy tissue, thus obtained, is calculated as a percentage of the original cocoa, and the result is multiplied by three, to give the percentage of husk present. This factor (3) was obtained after many estimations of heavy tissue in cacao husks. When husk is absent, the whole of the tissue is found floating on the surface of the medium, after the mixture has been centrifuged.

Finally, there is a method described by Wasicky and Wimmer\* for accurately estimating shell in ground cocoa, when less than 10 per cent. is present, by the use of ultra-violet light and a special form of microscope, made by Reichert of Vienna. The method is based on the fact that, when examined in ultra-violet light, powdered cacao bean appears violet-blue, whilst powdered shells have a brownish appearance.

0.01 gramme of cocoa is treated with 5 ccs. of mixed alcohol and glycerol for one hour; the mixture centrifuged, and the

\* R. Wasicky and C. Wimmer, *Zeitsch. Nahr. Genussm.*, 1915, xxx., 25.

liquid replaced by 1 cc. of a solution of borax and glycerol. The mixture is well stirred, and one drop is examined under the microscope, on a glass slide protected by a cover-slip. Cacao nibs and its paste give fine violet-blue colouration, the shell appearing brownish. The shell also shows a mucilaginous tissue of a colour varying between dull white and yellow-green.

By comparing the sample against standard slides, 1 per cent. of shell can, it is claimed, be readily detected.

### Chemical Methods.

It is impossible, in this place, to consider all the chemical methods suggested for estimating husk in cocoa. There has, however, been much recent work on the subject which cannot be discussed without a certain amount of retrospection.

Ulrich,\* in 1911, surveyed the then-existing methods for determination of fibre, with the following conclusions:—

(1) Microscopical method. Uncertain, owing to the fine comminution of modern cocoa powders.

(2) Elutriation method of Filsinger and Drawe (*Zeit. öffent. Chem.*, 1899, v., 27, and 1903, ix., 161). Fairly accurate only when the nib and shell have been ground together, the resulting particles of the latter being considerably larger than those of the former. Ulrich's conclusions have been considered earlier in this chapter.

(3) Flotation method in a solution of calcium chloride (Goske's method). Valueless.

(4) Chemical methods: (a) Proportion of shell and nib soluble in silicic acid (*Zeitsch. Nahr. Genussm.*, 1906, xii., 95, and *Zeit. öffent. Chem.*, 1908, xiv., 166); (b) Alkalinity of ash and content of total albumin (*Jour. Soc. Chem. Ind.*, 1905, 341)\*; (c) Iodine value of the fat (*Zeit. öffent. Chem.*, 1901, vii., 500); (d) Content of crude fibre or cellulose (*Jour. Soc. Chem. Ind.*, 1890, 421, 978; 1892, 344; 1906, 1069;

\* C. Ulrich, *Arch. Pharm.*, 1911, ccclix., 524—537.

*Zeitsch. Nahr. Genussm.*, 1898, iii., 1; 1908, viii., 769; 1906, xii., 159; 1907, xiii., 265; *Schweiz. Woch. Chem. Pharm.*, 1902, 462; (e) Content of pentosan and methyl-pentosan (*Jour. Soc. Chem. Ind.*, 1901, 396; 1902, 875; 1903, 114). All these methods are described by Ulrich as either valueless or not sufficiently delicate to detect relatively small proportions of shell. Ulrich's proposed new method did not, however, carry us much further for, as far as can be judged from his figures, only 10 per cent., or upwards, of added husk could be determined. His method, which is based on the fact that "cacao-red" is found only in the nib, is as follows:—

One gramme of the material, freed from moisture and fat, and very finely powdered, is boiled for three hours with 120 ccs. of acetic acid (50 to 51 per cent.) under a reflux condenser. After cooling, the mixture is made up to 150 ccs., at 15°C., with water, well shaken and allowed to stand for at least twelve hours. The liquid is then filtered through a dry filter-paper into a dry flask, and 135 ccs. (equivalent to 0.9 gramme of the original material) of the filtrate are mixed with 5 ccs. of concentrated hydrochloric acid and 20 ccs. of a 20 per cent. solution of ferric chloride. The mixture is boiled for ten minutes under a reflux condenser, rapidly cooled and

Cocoa.	Average amount of ferric chloride precipitate.					
	From roasted nibs, calculated on—			From unroasted nibs, calculated on—		
	Number of analyses.	Dry, fat-free material.	Original nib.	Number of analyses.	Dry, fat-free material.	Original nib.
		Per cent.	Per cent.		Per cent.	Per cent.
Guayaquil	12	16.11	7.479	3	16.67	7.312
Trinidad	12	14.11	6.143	3	—	—
Accra	6	13.82	6.337	3	14.82	6.294
Araba	18	13.50	6.998	3	12.82	5.842
Bahia	14	11.89	5.478	3	12.40	5.591
Ceylon	6	13.78	6.450	—	—	—
Samoa	12	14.34	6.698	3	14.90	6.492
S. Thomé	18	13.74	6.119	3	13.26	5.913

left for at least six hours, when the precipitate is collected on a weighed filter, washed with hot water, till free from iron, dried for six hours at  $105^{\circ}$  C. and weighed, the weight being calculated as a percentage of the dry, fat-free substance.

Ulrich found that, with the exception of Guayaquil cacao nibs which gave high results, and of Bahia cacao nibs which gave low figures, considerable uniformity existed in the amount of precipitate, obtained. This is shown in the above table of results.

Ulrich, however, relied upon greater uniformity in cocoa powders, which are seldom made from one quality of nibs only.

In 1912, Greifenhagen \* discussed the filtration of the hot solution, in König's method of crude fibre estimation, through a special asbestos filter by the aid of suction. Grossfeld,† in 1914, however, preferred filtration of the glycerol-sulphuric acid mixture through asbestos in a Gooch crucible without suction, as the fine particles tended to penetrate the layer of asbestos.

In 1916,‡ the following official German methods were published for the detection of excess of cacao shell in cocoa powders :—

*Moisture*.—Five grammes of the cocoa powder are mixed in a flat basin with 20 grammes of ignited sand and dried at  $105^{\circ}$  C. to constant weight, or nearly so, but the drying should not be prolonged more than four hours.

*Fat*.—Five grammes of the cocoa are placed in an asbestos filter-tube, 4 cms. in diameter (the asbestos is supported on a small filter-plate having holes about 1 mm. in diameter); the lower end of the tube is inserted into a hole in the rubber stopper closing a filter-flask, and the filter tube is filled with ether. As soon as the ether begins to drop into

\* W. Greifenhagen, *Zeitch. Nahr. Genussm.*, 1912, **xxiii.**, 101.

† J. Grossfeld, *Zeitch. Nahr. Genussm.*, 1914, **xxviii.**, 533.

‡ *Chem. Zeit.*, 1916, **xl.**, 969, extracted by *Jour. Soc. Chem. Ind.*, 1916, **xxv.**, 1269.

the flask, suction is applied, a further quantity of ether is added, and so on, using about 10 ccs. of ether each time, until 100 ccs. of ethereal extract have been collected. This ethereal solution is evaporated, and the residue of fat weighed.

*Crude Fibre.*—The residue remaining in the filter-tube is freed from ether, and, together with the asbestos, is transferred to a flask and boiled for one hour under a reflux condenser with 200 ccs. of 1·25 per cent. sulphuric acid. The mass is then collected on an asbestos filter, washed with hot water, again transferred, with the asbestos, to the flask, boiled for one hour with 200 ccs. of 1·25 per cent. potassium hydroxide solution, then collected on an asbestos filter and washed. This treatment with acid and alkali is repeated. The mass of crude fibre, obtained finally, is washed with alcohol and ether, transferred to a platinum basin, dried at 105° C. and weighed. The contents of the basin are then ignited, and the residue again weighed; the difference between the weights gives the crude fibre. The asbestos, used for the filters, should be treated previously with boiling dilute sulphuric acid and, later, with dilute potassium hydroxide solution, washed, dried and ignited. If the amount of crude fibre, found, exceeds 6 per cent., calculated on the dry, fat-free cocoa powder, the sample contains a quantity of shell in excess of that which should be present.

*Phosphates.*—Twenty grammes of cocoa are incinerated at a low temperature, the carbonised mass is collected on a filter, and its contents are ignited to a white ash, to which the filtrate is added and evaporated to dryness. After gentle ignition, the residue is treated with a few drops of hydrogen peroxide and 10 ccs. of 25 per cent. hydrochloric acid; the mixture is evaporated to dryness, the residue treated with hydrochloric acid and hot water, and the solution filtered. The filtrate is nearly neutralised with sodium hydroxide



solution, using methyl orange as indicator, then heated on a water-bath for 5 minutes, a further small quantity of alkali being added, but so that the solution still remains slightly acid, and the precipitated iron and aluminium phosphates are collected on a filter; the filtrate and the washings are diluted to 100 ccs. To determine the soluble phosphates, 10 ccs. of this solution are mixed with 30 ccs. of neutral 40 per cent. calcium chloride solution, a few drops of phenolphthalein solution are added, and the mixture is titrated with  $\frac{N}{10}$  sodium hydroxide solution, until a distinct red colouration is obtained; the mixture is now maintained at 15° C. for two hours, and the slight excess of alkali is titrated. Under these conditions, 1 cc. of  $\frac{N}{10}$  alkali solution is equivalent to 4.75 mgrms. of  $\text{PO}_4$ .

The insoluble phosphates are determined as follows:—Thirty ccs. of trisodium citrate solution (200 grms. of the salt dissolved in 300 ccs. of water) are placed in a flask, cooled in ice-water and, then, exactly neutralised, using phenolphthalein as indicator. The filter, containing the precipitate of iron and aluminium phosphates, is introduced into the flask, the mixture is heated on a water-bath for 20 minutes, then cooled in ice-water and titrated with  $\frac{N}{10}$  sodium hydroxide solution; in this case, 1 cc. of the alkali solution is equivalent to 9.5 mgrms. of  $\text{PO}_4$ . The results of both determinations are calculated into percentages of the cocoa powder. With a cocoa powder free from excess of shell, the quantity of insoluble  $\text{PO}_4$  will not exceed 4 per cent. of the total  $\text{PO}_4$ .

In 1917, Keller\* surveyed the various methods of estimating shell in cocoa powders, since he found that all cocoas, examined by him during the last six months,"

\* O. Keller, *Arch. Pharm.*, 1917, colv., 405—416.

had been adulterated with husk. This worker devised a method based upon his observation that the ethereal extracts of pure cacao nibs were colourless or faintly yellow, whilst the extracts of the husks were distinctly brown (*Apoth. Zeit.*, 1915, 560). His method is briefly as follows:—

Two grammes of the cocoa powder and 15 ccs. of ether are shaken together for twenty-four hours in a well-stoppered vessel at the ordinary temperature, the shaking being discontinued during the last two or three hours. Ten ccs. of the supernatant liquid are transferred to a test-tube and filtered through kieselguhr, repeatedly, if necessary, until a clear filtrate is obtained, into one comparison tube of a colorimeter. The test-tube and filter are washed with a quantity of ether, so that, after the washings have been added to the solution in the comparison tube, the liquid has a depth of 5 cms. The second comparison tube contains water, having a depth of 5 cms., to which is added ferric chloride (the liquid must be freshly prepared from official liq. ferri sesquichlor. and contain 0.1 gramme of iron in 100 ccs.) until the colours of the liquids, viewed from above, have the same strength. The comparison is repeated, with the difference that the quantity of water is diminished by a volume equal to that of the ferric chloride solution added. The volume of ferric chloride solution, required, increases with the husk-content of the powder. Pure cacao nibs, containing 54 per cent. of fat, require 2.4 ccs. of the ferric chloride solution; pure husk-free powder requires 1.4 ccs.; pure husks require at least 3.5 ccs., the average value being 4.4 to 4.5 ccs. The author was of the opinion that powders, containing 20 per cent., or more, of fat and requiring 2.5 ccs., or more, of the ferric chloride solution, had been adulterated with husk.

#### Estimation of Fibre.

Baker and Hulton estimated the fibre by treating the defatted material with 1.25 per cent. solutions of acid and

alkali, and made use of the following formula for calculating the percentage of shell in the sample :—

$$S = \left( \frac{K \times 160}{100 - (F + W)} - 5.7 \right) \times \frac{100 - (F + W)}{11.1}.$$

where  $S$  = percentage of shell on sample.  
 $K$  = percentage of fibre on sample.  
 $F$  = percentage of fat on sample.  
 $W$  = percentage of water on sample.  
 $5.7$  = percentage of fibre in dry, fat-free nib.  
 $16.8$  = percentage of fibre in dry, fat-free shell.

### Estimation of Nitrogen.

The nitrogen figure was estimated by the Kjeldahl-Gunning method on 1 gramme of the sample, the following formula being used :—

$$S = 2.17 \left( 100 - (F + W) \right) - \frac{100N}{2.26},$$

where  $S$  = percentage of shell on sample.  
 $N$  = percentage of nitrogen on sample.  
 $F$  and  $W$  as before.  
 $4.9$  = Nitrogen in dry, fat-free nib.  
 $2.64$  = Nitrogen in dry, fat-free shell.

The conclusions reached by Baker and Hulton were in favour of the levigation method, showing once more the diversity of opinion among the analysts.

Fallenberg,\* in 1918, did not further our knowledge to any great extent. This experimenter estimated the crude cellulose, free from lignin, and found in cacao ribs of different origin from 3.68 per cent. to 5.27 per cent., with an average of 4.44 per cent. Cacao husk yielded 9.5 per cent. to 22.4 per cent., with an average of 18.3 per cent., and it was considered by Fallenberg that cocoas yielding more than 5.5 per cent., calculated on dry, fat-free material should be

\* J. Fallenberg, *Mitt. Schweiz. Gesund.*, 1918, 277.

considered to be adulterated. The method he employed was to take 1 gramme of cocoa or 2 grammes of chocolate, extract with ether and dry in the water-oven. The powder was mixed with 30 ccs. of nitric acid and added to 120 ccs. of boiling nitric acid, and the whole boiled for ten minutes, with constant stirring. The mixture was filtered through a Gooch crucible lined with asbestos, and the residue washed, successively, with hot water, 1 per cent. solution of sodium hydrate, hot water, hot nitric acid, water and ammonia solution. Finally, the residue was washed with hot water, twice with alcohol and with ether, dried for an hour in the water-oven and weighed. The weight of the ash in the residue was deducted from the total, and the result was considered to be cellulose, free from lignin.

Still more recently, Hoepner\* has considered the question, and we are of the opinion that his conclusions are more nearly in the direction of solution of the problem than those who rely upon mechanical separation. The broad generalisation of his conclusions, however, rather mar his results.

Hoepner has stated that, if the crude fibre exceeds 6 per cent., the iron oxide 0.1 per cent., and the dry acid-insoluble ash 0.5 per cent., all calculated on the dry, fat-sugar-free material, the cacao product contains an excess of shell. The example given is of a sample containing 10.1 per cent. of crude fibre. The difference between 6 per cent. and this figure [ $= 4.1$ ], multiplied by 10, gives the actual excess of shell present, *i.e.*, 41 per cent.

It will be seen that such a method depends upon a standard method for estimation of crude fibre, and, until this has been done, we cannot see much hope of obtaining consistent results. We agree entirely with Cribb's remarks, to be found at the conclusion of Baker and Hulton's paper: "I am still of the opinion that the most satisfactory method

\* K. Hoepner, *Zeitsch. Nahr. Genussm.*, 1919, xxxvii., 18-31.

of ascertaining the proportion of shell in cocoa or *vice versa*, is by taking into consideration as many estimations of the constituents bearing on the problem as possible. No single estimation affords sufficiently reliable information. Though the levigation process may, with further experience, prove to be of the greatest value, it has become, in my opinion, less accurate since the present custom of grinding cocoas so finely came into vogue."

## APPENDIX

### PROVISIONAL DEFINITIONS AND STANDARDS OF CACAO AND ITS PREPARATIONS \*

#### (1) PURE CACAO MASS, PASTE, OR BITTER CHOCOLATE.

To consist of the crushed nibs of roasted and husked cacao beans only; it must not have been treated with alkalis or have been partially defatted.

To contain not less than 45 per cent. cacao butter.

„ „ „ more than 4 per cent. ash or mineral matter.  
(Ratio of soluble to insoluble ash  
not to exceed 2 : 3).

„ „ „ „ „ 6 per cent. moisture.

„ „ „ „ „ 2.75 per cent. pentosans.

„ „ „ „ „ 13 per cent. starch (which must be  
ascertained to be due to the  
cacao only, by means of the  
microscope).

To be free from added or included husk, cacao butter  
substitutes, starch, sugar, flavouring matter, etc.

#### (2) PURE COCOA POWDER.†

To consist of partially defatted, pure cacao mass only.

To contain not more than 6 per cent. of moisture.

\* The provisional standards, given herein, are essentially the same as those proposed by the author at the Congress of Cocoa and Chocolate Makers, held at Berne, in August, 1911.

† All cocoa and chocolate preparations (exclusive of cacao mass, paste, or bitter chocolate and cacao butter) may contain small quantities of harmless flavouring matters, which shall not include alkalis and other mineral chemicals.

To contain not more than 5 per cent. of ash or mineral matter (to show an alkalinity not exceeding 3 per cent. as  $K_2O$  on the dry, fat-free cacao matter).

3.5 per cent. pentosans.

5 per cent. starch (which must be ascertained to be due to the cacao only, by means of the microscope).

To be free from added or included husk, alkali, starch, sugar, etc.

### (3) "SOLUBLE" COCOA POWDER.

To consist of "pure cocoa" powder (as above) treated with alkali.

To conform with the standards of pure cocoa powder, with the exception that the ash-limit may be increased to 7 per cent., though the alkalinity may not exceed 5 per cent. estimated as  $K_2O$  on the dry, fat-free cacao matter.

### (4) PURE CACAO OR COCOA BUTTER.

To consist of the expressed fat of pure cacao mass only, or, if extracted from the husk, to be so declared.

To conform with the British Pharmacopœia standard of purity, and to give chemical and physical constants falling within the following limits:—

Zeiss butyro-refractometer value at 40° C.	46°—47°
Saponification value	192°—195°
Iodine value	34°—42°
Reichert-Meissl value	0.2°—0.9°

To be free from all animal and foreign vegetable oils and fats.

## (5) CHOCOLATE POWDERS.

The word "pure" to be applied only to those chocolate powders which contain pure cacao mass and fine crystallised sugar without further additions.

A removal of a portion of the fat from the cacao mass should not be allowed.

*Pure Chocolate Powders.*

To contain not more than 70 per cent. of sugar.

„ „ „ „ less than 30 per cent. pure cacao mass  
(estimated to contain 50 per cent. cacao butter).

The proportions of moisture, ash, starch, pentosans, etc., will be in accordance with the amount of cacao matter used.

If starch is added, the powder must not be labelled "pure," and the nature and quantity of the starch, added, should be clearly stated on the label, wrapper, etc. The extent of defatting of the cacao mass (if such has been made) should also be stated.

## (6) SWEETENED CHOCOLATE.

The term "chocolate" (with or without the word sweetened") to be applied only to those preparations which consist of pure cacao mass, fine crystallised sugar, and additional cacao butter (if desired) only.

To contain not more than 65 per cent. of sugar.

„ „ „ „ less than 32 per cent. of pure cacao mass  
(estimated to contain 50 per cent. cacao butter).

The proportions of moisture, ash, starch, pentosans, etc., will be in accordance with the amount of cacao matter used.

To consist of pure cacao mass, finely divided sugar and the solids of milk only.



The solids of the milk should be obtained from full-cream milk, and should be free from antiseptics.

To contain not less than 15 per cent. of milk solids.

" " " " " 3.75 per cent. of milk fat.

" " " " " 5.50 per cent. of milk sugar.

" " " " " 3.75 per cent. of protéids (over and above that due to the cacao matter present).

If no other fats than cacao butter and milk fat are present, the fat, extracted from a milk chocolate containing 25 per cent. milk solids, 10 per cent. cacao mass (containing 50 per cent. cacao butter), 15 per cent. added cacao butter, 50 per cent. cane sugar, will show the following chemical and physical constants:—

Zeiss butyro-refractometer value at 40° C. . . . . 45.5

Saponification value . . . . . 199.4

Iodine value . . . . . 85.6

Reichert-Meißl value . . . . . 9.23

#### (8) COVERING-CHOCOLATE.

To consist of variable quantities of pure cacao mass, finely crystallised sugar and additional cacao butter only.

#### (9) NUT, MALT, AND OTHER FANCY CHOCOLATES.

To consist of variable quantities of pure cacao mass, sugar, nuts, malt, etc., and to bear a label indicating the nature of such additions.

#### (10) CHOCOLATE BONBONS.

To consist of "fondants," "fruit jellies," etc., covered with pure covering-chocolate.

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# INDEX

- ACCRA cacao, characteristics of, 106, 111
- Accumulators for cacao butter press, 172
- Acetic acid fermentation, 71-74, 88
  - in cacao, 63, 65
- Added husk, estimation of, 533-548
- Adulteration of cacao preparations, 259, 262-266
  - detection of, 434-548
- Advantages of fermentation, 58-62, 70-74
- Acodoma cephalotes*, 54
- African cacao, characteristics of, 104-107, 111
  - output of, 21, 104-107
- Agriculture of cacao, 38-57
- Air-extracting machine, 238
- Albizzia, as shade for cacao, 50
- Albuminoids, or proteid matter, in husk, 397
  - in kernel, 409
  - estimation of, 509
- Alcohol from cacao pulp, 74-76
- Alcoholic fermentations, 70-74
- Alkalinity of commercial cocoa powders, 185, 187, 188, 192
  - estimation of, 482
  - provisional regulations concerning, 187, 183
- Alkali, application of, 176-182
- Alkalised chocolate, 283
  - cocoa powders, 175-183
- Alkaloids, estimation of, 498
  - present in cacao, 395-397, 410
- "Alligator" cacao, 32
- Amplonado cacao, 35
- American cacao, characteristics of, 92-99
  - output of, 21, 92-99
- "Amorphous" sugar, 232-236, 443, 447
- Analyses of African nibs, 121, 138, 149
  - shells, 121
- Analyses of American chocolates, 268, 298
  - ash of cacao husks, 393, 394
  - kernels, 148, 407, 408
  - Bahia nibs, 121, 138, 149
  - beans before and after fermentation, 62, 65
  - Caracas nibs, 121, 122, 136, 138, 149
  - shells, 124
  - Carupano nibs, 121
  - Ceylon nibs, 138
  - shells, 139
  - chocolate covering, 327, 328
  - "fondant," 267, 268, 271
  - milk, 290-301
  - plain, 261-273
  - powders, 273, 274
  - cocoa powders, plain, "soluble," and dietetic, 185-200
  - condensed milk, 319
  - date chocolate, 306
  - germ, 125, 148, 421
  - Grenada nibs, 121, 138
  - Guayaquil nibs, 121, 136, 138
  - shells, 139
  - husks of raw cacao, 124, 125, 372
  - kernels (nibs) of raw cacao, 119-125
  - of roasted cacao, 136-139, 400, 401
  - Java nibs, 121
  - Martinique nibs, 139
  - milk powder, 318, 319
  - Para nibs, 121
  - shells, 380
  - Puerto Cabello nibs, 137, 139
  - shells, 124
  - roasted cacao, 139, 375
  - soil, 40

- Analyses of Surinam nibs, 112, 137  
 — — — — — Trinidad nibs, 121, 136, 137  
 — — — — — shells, 124  
 Analysis, methods of, of albuminoids, 509  
 — — — — — ash or mineral matter, 480  
 — — — — — (soluble), 480  
 — — — — — (insoluble), 481  
 — — — — — cacao matter, 527  
 — — — — — cacao-red, 497  
 — — — — — caffeine, 498  
 — — — — — cane sugar, 517  
 — — — — — cellulose, 493  
 — — — — — crude fibre, 489  
 — — — — — fat, 448-478  
 — — — — — fibre, 488  
 — — — — — lactose, 520  
 — — — — — milk sugar, 520  
 — — — — — moisture, 479  
 — — — — — nitrogen, 509  
 — — — — — organic acids, 502  
 — — — — — oxalic acid, 503  
 — — — — — pentosans, 494  
 — — — — — reducing matters, 507  
 — — — — — starch, 511  
 — — — — — tartaric acid, 503  
 — — — — — theobromine, 498  
 — — — — — water extract, 487  
 — — — — — xanthine bases, 498  
 Ants, parasol, 54  
 Aphides, 55  
 Aroclor, methods of obtaining, 341-353  
 — — — — — of different caçaos, 92-108  
 Arriba, Guayaquil cacao, characteristics of, 92  
 Ash from cacao, composition of, 148, 393, 394, 407, 408  
 — — — — — from cocoa powders, composition of, 185, 190, 192, 419-423  
 Asiatic cacao, characteristics of, 107, 108  
 — — — — — output of, 21, 107, 108  
 Automatic chocolate weighing machine, 228, 251-257  
 Average dimensions of cacao beans, 116  
 — — — — — weights of cacao beans, 110, 111  
 BACTERIA, in fermentation of cacao, 70, 88  
 Bahia cacao, characteristics of, 97, 111, 113, 114  
 — — — — — output of, 97  
 Bananas, as catch crop, 51  
 Bark, precautions concerning, 51-57  
 Bassia tallow, physical and chemical constants of, 478  
 Bastard vanilla, 348  
 Battery of cocoa butter presses, 170-172  
 — — — — — of refiners, 207, 210  
 — — — — — of tapping tables, 246  
 "Bausman Disc" Reducer, 228  
 — — — — — Refiner, 229  
 Beans, analyses of cacao. See *Analyses*.  
 Beef fat, physical and chemical constants of, 478  
 Beeswax, physical and chemical constants of, 478  
 Beetles, cacao, 54  
 Belgian Congo cacao, output of, 21, 107  
 Bensdorp cocoa, analysis of, 188, 192  
 Benzoin gum, as varnish for chocolate, 353  
 Berbice cacao, characteristics of, 96  
 Bibliography, 553-555  
 Biscuit covering, 324-340  
 Bitter chocolate, 167  
 Bjorklund's ether test, 457  
 Black rot, 51  
 Blocker's cocoa, analysis of, 187, 188, 190  
 "Bloom" on chocolate, 260  
 Bois immortel, 49  
 Books referred to, 553  
 Borneo tallow, physical and chemical constants of, 478  
 Botanical description, 33  
 "Bramigk" ball machine, 231  
 Brazil cacao, characteristics of, 96  
 — — — — — output of, 21, 96  
 Breaking pods of cacao, 64  
 Butter, cacao. See *Cacao butter*.  
 — — — — — press, 170  
 Butter fat, physical and chemical constants of, 478  
 Cacao, Accra, characteristics of, 106, 111  
 — — — — — African, characteristics of, 104-107, 111  
 — — — — — agriculture of, 38-57  
 — — — — — American, 92-99  
 — — — — — American imports and exports of, 22  
 — — — — — as money, 3

- Cacao, Bahia, characteristics of, 97, 111, 114
  - output of, 97
  - bark, use of, 51-57
- beans, chemical constituents of, 119-125, 371-416
- bean sorter, 132
- beetles, 54
- Berbice, characteristics of, 96
- blends of, 276, 281, 282
- botany of, 31-37
- Brazil, characteristics of, 96
  - output of, 21, 96
- breaking, 64
- butter, adulterations of, 474
  - chemical constituents of, 381
  - definitions of, 550
  - digestibility of, 364
  - estimation of, 448-478
  - expression of, 169
  - filtration of, 172
  - in chocolate, 265, 266, 268, 271, 291-296, 302, 327, 328
  - in cocoa powders, 184-195, 423
  - in husks, 381-390
  - in ribs, 403-406
  - iodine value of, 385
  - melting point of, 384, 402
  - method of determining melting point of, 461
    - physical and chemical constants of, 457-478
  - physical and chemical constants of, 381-390, 478
  - press, 140
  - Reichert-Meis value of, 389, 478
  - saponification value of, 387
  - specific gravity of, 383, 460
  - substitutes, 474
  - tests for adulteration of, 457-478
- Cameroon, characteristics of, 104
  - output of, 21, 104
- Caracas, characteristics of, 96
- Ceylon, characteristics of, 107
  - output of, 21, 107
- cleaning and sorting machine, 111
- colouring matter from, 65, 398, 411
- consumption of, 18-20, 29
- Cuba, characteristics of, 97
  - cultivation of, 38-57
  - description of commercial, 99-117
  - diseases of, 51-55
  - drying, 78
  - English imports and exports of, 24-27
  - Esmeraldas, characteristics of, 92
  - Essequibo, characteristics of, 96
  - extract of, as improver, 378
  - fermentation of, 58-76
  - finishing, 80-85
  - first French crop of, 10
  - flowers of, 34
  - food-value of, 354-367
  - fruit of, 34
  - germ, analyses of, 125, 148, 421
  - germ-extracting machine, 154
  - glucoside of, 412
  - Grenada, characteristics of, 101
    - output of, 21, 101
  - Guayaquil Arriba, characteristics of, 92
    - Machala, characteristics of, 92
  - history of, 3-17
  - husk as fodder, 377
    - as manure, 378
  - husking and winnowing machine, 150
  - husks, analyses of, 124, 125, 130, 380
  - Jamaica, characteristics of, 102
    - output of, 21, 102
  - Java, characteristics of, 108
    - output of, 21, 108
  - kernels, analyses of, 119-125, 136-139, 400, 401
  - manures and manuring, 41-46
  - Maracaibo, characteristics of, 94
  - Maranhão, characteristics of, 96
  - markets for, 21-29
  - mass (see also *Analyses of roasted kernels*), 162-169
  - matter, estimation of, 292, 527
  - microscopy of, 434-445
  - mills, 163
  - mythology of, 4
  - picking, 64
  - plantations, 39, 48
  - planting, 48
  - planting of, ancient ceremony, 6
  - polishing, 84
  - powder. See *Cocoa powder*.



- **Cacao**, pruning trees of, 55
  - red, 65, 398, 411
  - — estimation of, 497
  - roaster, 142
  - San Domingo, characteristics of, 100
    - — — output of, 21, 100
  - San Thomé, characteristics of, 104
    - — — output of, 21, 104
  - Soconusco, characteristics of, 99
  - shade trees, 49
  - shelling, 64
  - soils, 40
  - statistics, 18-30
  - Surinam, characteristics of, 95
    - — — output of, 95
    - — — theobromine from, 395, 410
    - — — tree, 31, 34
  - Trinidad, characteristics of, 101
    - — — output of, 21, 101
  - — — varieties of, 35
  - Venezuela, characteristics of, 93
    - — — output of, 21, 93
    - — — washing, 77
  - West Indian, characteristics of, 99
    - — — — output of, 21, 99
- Cacaonin, 413
- Cadbury's cocoa, analysis of, 186, 187, 192, 359-362
  - — — digestibility of, 359-362
- Caffeine in cacao beans, 395, 411
  - — — estimation of, 498
- Calabacillo cacao, 35-37, 65, 87, 120
- Cameroon beans, characteristics of, 104
  - — — fermentation, data of, 68
  - — — output of, 21, 104
- Cancerous parasites, 55
- Cane sugar as adulterant in cocoa powders, 188, 197, 198
  - — — in chocolate, 265-273, 276-283, 429
  - — — estimation of, in chocolate, 517
- Canker, 51
- Caracas beans, analyses of, 121, 122, 124
  - — — characteristics of, 93
- Caramelising machine, 346
- Carupano cacao, characteristics of, 94
- Casein, estimation of, 530
- Cassava, as catch crop, 51
- Castilloa elastica, as catch crop, 50
- Catch crops (for cacao plantations), 49-51
- Cellulose in cacao beans (see also under *Analyses*), 421, 406
  - — in cocoa powder (see also under *Analyses*), 426
  - — in husk, 393
  - — — estimation of, 493
- Ceylon cacao, characteristics of, 107
  - — — fermentation, data of, 67, 68
  - — — output of, 21, 107
- Chambers, cooling, for chocolate, 248
  - — — heating, for chocolate, 215
- Characteristics of cacao varieties, 90-118
- Chillies, as catch crop, 51
- Chinese tallow, physical and chemical constants of, 478
- Chocolate air-extracting machine, 238
  - — as inflamer of passions, *Introduction*, 7, 357
  - — — automatic weighing machine for, 251-256
  - — — "conche" machine, 322
  - — — cooling machines, 248
  - — — covering, analyses of, 327, 328
  - — — recipes for making, 327
  - — — croquettes, 247
  - — — definitions of, 549
  - — — derivation of word, 31
  - — — drop press, 247
  - — — early monopoly of, 10
  - — — first appearance in Europe of, 7
  - — — "fondant," 284, 307-323
  - — — food-value of, *Introduction*, 355-367
  - — — forcing and weighing machine, 251-256
  - — — improvers, 352, 379
  - — — "mélangeur," 294
  - — — methods of analysing, 448-532
  - — — milk, analyses of, 290-301
  - — — recipes for making, 290-301
  - — — mixing and kneading machines, 204, 330
  - — — moulds, 244
  - — — mould-filling machines, 251-257
  - — — neapolitans, 247
  - — — nut, recipes of, 293
  - — — nutritive value of, *Introduction*, 355-367
  - — — plain, analyses of, 261-273
  - — — recipes for making, 276-287

- Chocolate powder, 287  
 — refiners, 206  
 — surrogate, 378  
 — tapping table, 245  
 — teas, 373-381  
 — temperature for moulding, 271-277  
 — tempering machines, 239  
 — varnish, 259, 353  
 Chocolaterie ouvrière cocoa, analysis of, 188  
 Citronella, as catch crop, 51  
 Classification of cacao, commercial, 90, 91  
 — — Hart's, 35  
 — — Morris', 35  
 "Claying" cacao, and washing, 77-85  
 Climate for successful cultivation, 39  
 Cocoa powder, adulterants of, 197  
 — analyses of, 185-200  
 — composition of, 417-427  
 — crusher and disintegrator, 173  
 — methods of analysing, 448-532  
 — plant for manufacturing, 183  
 — sifter, 173  
 — "soluble," recipes of, 175-183  
 Cocoa-red, in cacao, 398, 411  
 — estimation of, 497  
 Coconut fat, detection of, 457-478  
 — physical and chemical constants of, 478  
 Coffee, as catch crop, 51  
 Cold-water extract of cacao, etc., 430, 487  
 — — — determination of, 487  
 Colletotrichum, 52  
 Colouring cacao, 81  
 Colour of cacao fruit, 34  
 Commercial kinds of cacao, 90-118  
 "Conche" machine, 322  
 Condensed milk, preparation of, 311-314  
 — — for preparation of milk chocolate, 314  
 Consumption of cacao, etc., 29  
 Cooling machinery for tablets, etc., 248-255  
 — — for roasted beans, 142, 145  
 Conté cocoa, analysis of, 188  
 Continuous mould-filling and tapping machine, 251  
 Copper in the ash of cacao, 395  
 Cotton, as catch crop, 51  
 Cotton-seed saccharins physical and chemical constants of, 478  
 Coumarin, used as flavouring material, 352  
 Covering-chocolate, 324-340  
 Criollo cacao, 35  
 Croquettes, chocolate, 247  
 Cude fibre, estimation of, 480  
 — — in cacao (see also under *Analyses*), 390, 406  
 Crushers for cocoa powder, 173  
 Cuban cacao, characteristics of, 97  
 — — output of, 21, 97  
 Cultivation of cacao, 38-57  
 Cundeamor variety of cacao, 35  
 Curing cacao, 58-89  
 — — loss of weight during, 65, 72, 77, 109  
 Cuticles of beans, analyses of (see also *Analyses of husk*), 120  
 Damp cacao beans, cure for, 131  
 "Dancing" cacao, 84  
 Date chocolate, 305  
 Definitions of cacao and cocoa preparations, 549  
 Déjong's cocoa, analyses of, 188  
 Dekker's method for estimating theobromine, 500  
 Detection of added husk in cocoa powder, 480-497, 533-548  
 Digestibility of chocolate, 354-364  
 — of cacao butter, 364-366  
 Dika fat, physical and chemical constants of, 478  
 Diorite for chocolate refiners, 207  
 Diseases of cacao, 51  
 Distance in cacao planting, 43  
 Disintegrator for cocoa powder, 173  
 Distribution geographical, of cacao, 15, 20, 21  
 Dominica cacao, characteristics of, 103  
 — — output of, 21, 193  
 Domingo, San, cacao, characteristics of, 100  
 — — output of, 21, 100  
 Draining land for cacao, 39  
 Drop press for chocolate, 247  
 Dry varieties of chocolate, 277-283

- Drying of cacao beans, 78  
 Dryness, to maintain, in factory, 241-244, 250  
 Dust-removing machine, 132  
 Dutch cocoa, preparation of, 175-182  
 — Guyana cacao, output of, 96  
 — imports and exports, 20, 29  
  
 EARLY monopoly of chocolate, 10  
 Earth-nut oil, physical and chemical constants of, 478  
 Eating-chocolate, analyses of, 261-273, 290-301  
 — — — recipes of, 276-286, 290-304, 320, 327  
 Ecuador cacao, characteristics of, 92  
 — — — output of, 21, 92  
 Egg chocolate, 305  
 "Enrober," 336  
 "Enpholda," 340  
 Epps's cocoa, analyses of, 186, 192, 197  
 Esmeraldas cacao, characteristics of, 92  
 Essential oil of cacao, 79, 416  
 Essequebo cacao, characteristics of, 96  
 Essences for flavouring chocolate, 347  
 Ethereal oils as flavourings, 347  
 Examination, microscopical, of cacao, etc., 434-447  
 Exports of cacao, 20-29  
 Expression of fat, 169  
 Extraction of fat, 448-457  
 Erythrina, as shade for cacao, 49  
  
 FAT. See *Cacao butter*.  
 "Fatiguing" chocolate, 212  
 Fermentation, bacteria taking part in, 70  
 — chemical changes during, 65, 71  
 — loss during, 73  
 — temperature of, 68  
 Fernando Po cacao, characteristics of, 106  
 Fibre in cacao husk, 390  
 — — — nibs, 406  
 — — — estimation of, 486-497  
 Fiji cacao, output of, 107  
 Filling chocolate moulds, 237-257  
 Finishing cacao beans, 80  
 Flavouring materials for chocolate, 347  
 Flowers of cacao tree, 34  
 "Fondant" chocolate, method of preparation, 307, 320-323  
 — — — analyses of, 267, 268, 271  
 — — — recipes for making, 320  
 Forastero cacao, 35, 62  
 French chocolate, analyses of, 266  
 — — — recipes for, 267, 281  
 Fruit of cacao tree, 34  
 Fry's cocoa, analyses of, 186, 187, 192  
 Fungi on cacao fruit, 52  
 Fungicides for spraying, 53  
 Furfural, estimation of, for pentosans, 495  
  
 GALAM butter, physical and chemical constants of, 478  
 Gathering cacao, 56  
 Germ, cacao, analysis of, 125, 148, 421  
 — — — extractor, 154  
 German chocolate, analyses of, 266  
 — — — recipes for, 281  
 Germination of cacao, 48  
 Globulin in cacao, 397  
 "Gloor" patent, 231  
 Goa butter, physical and chemical constants of, 478  
 Gold Coast cacao, fermentation data of, 68  
 — — — output of, 21, 105  
 Grafting cacao, 47  
 Granite for cacao machinery, 207, 224-226  
 Gravity, specific, 388, 460  
 — — of cacao butter, etc., 384, 461, 478  
 — — — methods of determining, 460  
 Grenada cacao, characteristics of, 101  
 — — — output of, 21, 101  
 Grinding cocoa powder, 173  
 — mill for beans, 163  
 Ground-nuts, as catch crop, 51  
 Ground-nut oil, as adulterant of cacao butter, 478  
 Growth of cacao, rate of, 34, 56  
 — — — industry, rate of, 14-17  
 Guadeloupe cacao, characteristics of, 102  
 — — — output of, 21, 102  
 Guatemala cacao, characteristics of, 99  
 Guayaquil cacao, characteristics of, 92  
 — — — output of, 92

- Guayra cacao, 90  
 Guiana, British, cacao, characteristics of, 96  
 — — — output of, 21, 96  
 Gum benzoin, as varnish for chocolate, 353
- HARTI cacao, characteristics of, 99  
 — — — output of, 21, 99  
 Hand-covering, methods of, 331  
 Hart's classification of cacao, 35  
 Harvesting cacao, 56  
 Hazel-nut oil, physical and chemical constants of, 478  
 Hevea brasiliensis, as shade trees, 50  
 History of cacao, 4-17  
 Hot chambers, 215  
 Houten, Van, cocoa, analysis, 186-188, 190, 192, 359-362  
 Hubl's method of determining iodine value, 471  
 Husks, analyses of raw, 124, 125, 372, 375  
 — — — roasted, 139, 375  
 — as fodder, 377  
 — characteristic appearance of, of different cacaos, 92-109, 114, 117  
 — chemical composition of, 124, 125, 139, 372, 375  
 — detection of, in cocoa, chocolate, etc., 425, 480-497, 533-548  
 — extraction of fat from, 381, 448-457  
 — percentage of, in beans, 112, 113, 115, 372  
 — removal of, 150  
 — uses of, 377, 378  
 Husking machine, 150  
 Hydraulic press for cacao butter, 170
- Imports of cacao, etc., 20-29  
 Improvers, chocolate, 352, 379  
 Ingredients used for cocoa preparations.  
     See Recipes.  
 Insoluble ash, method of determining, 481  
 Inter- or catch-crops for cacao, 50, 51  
 Investigation, chemical, of cacao, etc., 58-76, 119-125, 372, 433, 448-548  
 — microscopical, 434-447
- Iodine values of cacao butter and its adulterants, 385, 471, 478  
 — — — methods of determining, 47.
- JAMAICA cacao, characteristics of, 102  
 — — — output of, 21, 102  
 Java cacao, characteristics of, 108  
 — — — output of, 21, 108  
 Journals referred to, 553-555  
 Judging cacao, 109
- KARITÉ fat, physical and chemical constants of, 478  
 Kernels, analyses of raw, 119-125  
 — — — of roasted, 136-139, 149, 400, 401  
 — changes in, during fermentation, 65, 72  
 — characteristics of different varieties of, 90-118  
 — chemical composition of, 119-125, 136-139, 400, 401  
 — methods of analysis of, 448-517  
 — proportion of, in beans, 112, 113, 115, 400  
 Kneading machine for chocolate, 237, 330  
 Koettstorfer value of fats, 387, 478  
 — — — methods of determining, 466  
 Kokum butter, physical and chemical constants of, 478  
 Kreuz method of estimating cacao butter, 452  
 Kunze's method of estimating theobromine, 499
- LACTOSE in milk chocolate, estimation of, 517-527  
 Land for successful culture of cacao, 38-46  
 Lard, physical and chemical constants of, 478  
 Lemon grass, as catch crop, 51  
 Levigation of chocolate, 206-215, 224-227  
 Lime, necessary for successful culture of cacao, 40  
 "Landt" flavour, 286

- Losses in preliminary preparation of beans, 115, 155-157  
 — on washing cacao, 77
- MACHALA, Guayaquil, cacao, characteristics of, 92
- Machine, air-extracting, 238  
 — automatic tempering, 239  
 — caramelising, 346  
 — "Champion" dipping, 333  
 — chocolate cooling, 248, 251-255  
 — cleaning and sorting, 132  
 — "conche," 322  
 — continuous mould-filling and tapping, 251-255  
 — covering, methods of, 331-340  
 — drop press, 247  
 — "Enrober," 336  
 — "Enpholda," 340  
 — forcing and weighing, 251-256  
 — germ extracting, 154  
 — milling, 160  
 — mixing, 204, 330  
 — mixing and kneading, 330  
 — mould-filling, 251-257  
 — nibbing, husking and winnowing, 150  
 Madre de Cacao, as shade trees, 49  
 Mafura, tallow, physical and chemical constants of, 478  
 Magnesium carbonate for "soluble" cocoa, 179  
 Malaba tallow, physical and chemical constants of, 478  
 Manihot, as shade tree, 50  
 Manufacture of chocolate, 112-169, 201-236, 307-323  
 — — cocoa powders, 169-183  
 — — condensed milk, 311-314  
 — — milk powder, 315-319  
 — — "soluble" cocoa powders, 175-183  
 Manures for cacao plantations, 41-46  
 — effect of, on yield, 45, 46  
 Maracábo cacao, characteristics of, 94  
 Maranhão cacao, characteristics of, 93  
 Markets for cacao, 22, 23  
 Martinique, first crop of cacao from, 10, 16  
 — cacao, characteristics of, 102  
 Materials, flavouring, for chocolate, 347
- Matter, cacao, estimation of, 527  
 — reducing, estimation of, 507  
 Mauritius cacao, output of, 21, 197  
 Meissl-Reichert value of fats, 389, 478  
 — — method of determining, 468  
 "Melangeur," 204  
 — use of, for chocolate making, 204-206  
 "Meltis" chocolate, analysis of, 267  
 Menier cocoa, analysis of, 188, 190  
 Melting points of cacao butter substitutes and adulterants, 478  
 — — of fatty acids, 464  
 — — methods of determining, of cacao butter, etc., 462  
 Mexican cacao, characteristics of, 99  
 Microscopical examination of cacao, 434-439  
 — — of chocolate, 439-447  
 Milk chocolate, analyses of, 290-301  
 — — components of, 430  
 — — definitions of, 551  
 — — methods of analysing (see also *Plain chocolates*), 520-532  
 — — milk solids in, estimation of, 529  
 — — recipes, 290-301  
 — fat, physical and chemical constants of, 478  
 — powder, for milk chocolate, 318, 319  
 — — preparation of, 315-319  
 — — report of L.G.B. on, 432  
 — sugar, estimation of, 520-527  
 Mills for grinding cacao beans, 163  
 Mineral matter, estimation of, 480  
 — — in husks, 394  
 — — in kernels, 467  
 "Misérables," composition of, 375  
 "Mitscherlich" bodies, 435  
 Mixture. See *Recipes*.  
 Moisture in cocoa preparations, 418  
 — methods of estimating, 479  
 Moreuil cocoa, analysis of, 188, 190  
 Morris' classification of cacao, 35  
 Moths, cacao, in store rooms, etc., 131  
 Mould-filling machine, 251-255  
 Moulding chocolate, 237-257  
 — temperature for, 244  
 Mouldy cacao, treatment of, 130  
 Mythology relating to cacao, 4

- NEAPOLITANS, moulding, 247
- Native Ceylon cacao, characteristics of, 107
- Neotria Reinii, 51
- Nibs, analyses of raw, 119-125
- of roasted, 136-139, 149, 400, 401
- methods of analysis of, 448-517
- Nigerian cacao, output of, 21, 106
- Nitrogen in cacao, 397, 409
- — husks, 397
- — nibs, 409
- — trees, 41
- estimation of, 509
- Nursery for cacao, 48, 49
- Nut chocolate, recipes for making, 303
- Nut oil, physical and chemical constants of, 478
- Nutmeg butter, physical and chemical constants of, 478
- Nutritive value of chocolate, *Introduction*, 354-367
- OCHRE, adulteration with, 262
- Oils, essential, for flavouring, 347
- Ordinary chocolate powder, recipe for making, 288
- milk chocolate, recipe for making, 300
- Organic acids, estimation of, 502
- Oxalic acid, estimation of, 503
- in cacao, 188, 413
- PACKING chocolate, 257
- Palm kernel oil, physical and chemical constants of, 478
- Palm oil, physical and chemical constants of, 478
- Para cacao, characteristics of, 96
- Para rubber as shade trees, 50
- Paraffin wax, physical and chemical constants of, 478
- Parasol ants, 54
- Pectins, utilisation of cacao, 46
- Pentacosane, estimation of, 494
- in cocoa powders, 426
- in husks, 391
- Pepper, as catch crop, 81
- Petroleum ether for extracting fat, 453, 456
- Phosphoric acid, 249-257
- Phulwara butter, physical and chemical constants of, 478
- Phytophthora Faberii, 31
- omnivora, 52
- Physical and chemical constants of cacao butter and substitutes, 478
- Picking cacao fruit, 56
- Pigment cells of cacao, 437
- Pine-nut oil, physical and chemical constants of, 478
- Piney tallow, physical and chemical constants of, 478
- Plain chocolate, analyses of, 261-273
- — recipes for making, 276-289, 290-301
- Plant acids, estimation of, 502
- Plant food removed from soil by cacao, 40
- Plasmon cocoa, digestibility of, 359-362
- Pods, description of cacao, 34
- illustration of, *Frontispiece*
- "Polishing" cacao, 84
- — automatic machines for, 85
- Possible adulterants of cacao butter, detection of, 457-478
- — physical and chemical constants of, 478
- Potash for rendering cocoa "soluble," 176-182
- Powder, cocoa, components of, 417-427
- — methods of analysis of, 448-548
- — "soluble," 175-183
- chocolate, components of, 428-433
- — methods of analysis of, 448-548
- — recipes for making, 287-289, 301, 303
- Powdering cocoa, 173
- Preparation of "chocolat fondant," 320-323
- — chocolate, plain, 201-236
- — cocoa powder, 169-183
- — flavours, 347-353
- — milk chocolate, 309-319
- — "soluble" cocoa powder, 175-183
- Preservation of cacao beans, 130-132
- Press, cacao butter, 170
- chocolate drop, 247
- Process of manufacture, influence of, 249-257

- Proportion of nibs and husks in cacao beans, 112, 113, 152, 158, 371-380, 400-402
- Proteid matter in cacao nibs, 395  
 — — — husk, 397  
 — — — estimation of, 399
- Provisional definitions and standards, 549
- Pruning cacao trees, 55
- Puerto Cabello cacao, characteristics of, 95
- Pulveriser, cocoa powder, 173
- QUANTITY and quality of beans from natural causes, 86-89  
 — — — from fermentation and curing, 58-76
- RADICLES, removal of, 154
- Raw cacao husks, analyses of, 124, 125, 372  
 — — — kernels, analyses of, 119-125
- Recipes, chocolate "fondant," 320  
 — — — powders, 287, 289, 301, 303  
 — — — covering-chocolate, 327, 328  
 — — — milk chocolate, 290-301  
 — — — plain eating-chocolate, 276-287  
 — — — vanilla sugar and essence, 348-350  
 — — — vanillin sugar and essence, 349
- Red, cacao-, in nibs, 411  
 — — — estimation of, 497
- Reducing matter in cacao, 507  
 — — — estimation of, 507
- Refiners, 206  
 — — — steel *versus* granite, 224-226
- Refining chocolate, 206-215
- Refractometer values of cacao butter substitutes and adulterants, 389, 478  
 — — — methods of determining, 458
- Refrigeration methods, 248-250
- Reichert-Meißl value of cacao butter and substitutes, 389, 478  
 — — — methods of determining, 468
- Résumé* of Chapters IV.-VIII., 86-89
- Ripeness of cacao fruit, determining, 57
- Roasted cacao husks, analyses of, 124, 125, 372
- Roasted cacao kernels, analyses of, 136-139, 400, 401
- Roasters for open fire, 142  
 — — — externally heated, 144
- Roasting cacao, 135-145
- Rowntree's cocoa, analyses of, 186, 189
- Royal Soconusco cacao, characteristics of, 99
- Rubber, as shade trees, 50
- SACCHARIN, 111-122
- Saccharin, for diabetic cocoa, 199
- St. Lucia cacao, characteristics of, 103  
 — — — output of, 21, 103
- St. Vincent cacao, characteristics of, 103
- Samana cacao, characteristics of, 100
- Samoa cacao, characteristics of, 107
- Sampling, during roasting of cacao, 142
- San Domingo cacao, characteristics of, 100  
 — — — output of, 21, 100
- San Thomé cacao, characteristics of, 101  
 — — — output of, 21, 104
- Saponification value of cacao butter substitutes and adulterants, 387, 478  
 — — — methods of determining, 466
- Seed, analyses of. See *Analyses*.  
 — — — planting, 48  
 — — — selection, 46
- Seychelles cacao, characteristics of, 107
- Shade plants for cacao trees, 49
- Shaking table, 245
- Shea butter, physical and chemical constants of, 478
- Shell butter, 378, 381
- Shelling cacao pods, 64
- Shells. See *Husks*.
- Short-cuts to chocolate manufacture, 217-236
- Sifting cocoa powder, 173
- Silent tapping table, 245
- Silicic anhydride in ash of cacao husk, 393, 394  
 — — — — nibs, 395, 396
- Site for cacao plantation, 39
- Soconusco, Royal, cacao, characteristics of, 99
- Soils for successful cultivation of cacao, 40

- Soluble, estimation of, 440  
 Soluble cocoa powder, chemical composition of, 185, 188, 192, 359-362, 418-427  
 — — — preparation of, 175-183  
 — — — properties of, 358, 360  
 Sorting cacao beans, 132  
 — — — machines, 132  
 Spanish chocolate, analyses of, 266  
 — — — recipes for making, 282  
 Specific gravity of cacao butter substitutes and adulterants, 383, 461, 478  
 — — — methods of determining, 460  
 Spices, used as flavouring materials, 347  
 Spiral bodies in cacao, 434  
 Standards of cacao, etc., provisional, 549  
 Starch in cacao, etc., 398, 404, 429  
 — — — estimation of, 511  
 — — — microscopical appearance of, 404  
 Statistics of cacao, 18-30  
 Tartaric acid in cacao butter, 381  
 Steel roll refiners, 206-215, 224-226  
 Steirastoma histrioides, 54  
 — — — depressum, 54  
 Storing cacao beans, 130-132  
 Substitutes for cacao butter, 474  
 — — — detection of cacao butter, 474  
 — — — physical and chemical constants of cacao butter, 478  
 Sugar consumption, statistics of, 217  
 — — — estimation of, 517-527  
 — — — in cacao nibs (see also *Analyses*), 427  
 — — — in chocolate (see also *Analyses*), 429  
 Sunlight, protection of cacao trees from, 49  
 Surinam cacao, characteristics of, 95  
 — — — fermentation, data of, 68  
 — — — output of, 95  
 Surrogate powder, 378  
 Survey of components of cacao husks, 391-398  
 — — — nibs, 400-416  
 — — — cacao preparations, 417-433  
 Sweetings of cacao, analyses of, 46  
 Sweetened chocolate, definitions of  
 TAPPING  
 Tartaric acid in cacao, 418  
 — — — estimation of, 502  
 Temperature, extreme, for culture of cacao, 39  
 — — — for fermenting cacao, 67-69  
 Temperature for moulding chocolate, 241  
 Theobroma augustifolia, 31  
 — — — bicolor, 31, 33  
 — — — cacao, 31-37  
 — — — ovatifolia, 31  
 — — — pentagona, 31, 32  
 — — — sylvestris, 31  
 Tibbles' cocoa, analysis of, 186  
 Treatment of defective cacao beans, 130-132  
 Trinidad cacao, characteristics of, 101  
 — — — fermentation of, data of, 68  
 — — — output of, 21, 101  
 Triple mill for cacao, 163  
 UGANDA  
 UGANDA cacao, output of, 21, 106  
 Unfermented beans, analyses of, 62, 65  
 — — — commercial value of, 64  
 Upright refiner, 207-215  
 VACUUM process for condensing milk, 311-314  
 — — — for milk powder, 315-318  
 Van Houten's cocoa, analyses of, 186-188, 190, 192, 359-362  
 Vanilla as flavouring for chocolate, 348  
 — — — characteristics of, 348  
 — — — sugar and essence, preparation of, 348  
 Vanillin as flavouring for chocolate, 349  
 — — — characteristics of, 349  
 — — — sugar and essence, preparation of, 349  
 — — — prices of, compared with vanilla, 349  
 Varieties of cacao tree, characteristics of, 31-33  
 Vegetable fats as substitutes for cacao butter, 474-478  
 — — — physical and chemical constants of, 478  
 Venezuela cacao, characteristics of, 93  
 — — — output of, 21, 93  
 Volatile alkalis for fendering cocoa "cocoa," 178, 184, 185



"Walker" machine 278  
 Wax, oil, physical and chemical con-  
 stants of, 478  
 Warming cupboard, 215  
 Washing cacao, advantages, etc., of  
 • 77-27, 230  
 — losses on, 77  
 Waste products in manufacture, 115,  
 155-157  
 Water chocolate, 278  
 Wax, bees, chemical and physical con-  
 stants of, 478  
 — paraffin, chemical and physical con-  
 stants of, 478  
 Weighing, automatic machine for choco-  
 late, 251-256

West African bases

104-107, 111

West Indian cacao

99-103, 112, 113

Wind shelter, cacao

Worms in cacao beans, curing, 153

XANTHINE bases, estimation of, in cacao  
 etc., 498

YIELD of nibs in manufacture, 115, 130,  
 155-157

